

# The Chemical Age

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## Contents

	PAGE
EDITORIAL NOTES: 1929—A Year of Progress Through Unsettled Conditions .....	585
A Review of 1929: British Chemical Industry in 1929, by Sir William Alexander; Recent Progress in the Gas Industry, by Sir D. Milne-Watson; British Chemical Industry—A Review of the Past Year, by J. Davidson Pratt; The Nitrogen Industry in 1929, by Dr. E. B. Maxted; Chemical Research Undertaken by the D.S.I.R.; The Synthetic Fertiliser Industry; The British Dyestuffs Industry in 1929—(1) Technical Progress, by L. J. Hooley—(2) The Colour User's Point of View; The Year's Progress in the British Solvents Industry; Progress Among Chemical Plant Manufacturers, by J. Arthur Reavell; Conditions in the Heavy Chemical Industry in 1929, by P. Parrish; A Review of the Year's Chemical Inventions; The Association of British Chemical Manufacturers .....	588
Chemical and Allied Societies; Their Work During the Year .....	619
The Dyestuffs Patent Action .....	625
Scottish Coal Tar Products During 1929 .....	626
Patent Literature .....	628
Commercial Intelligence .....	632

**NOTICES:**—All communications relating to editorial matter should be addressed to the Editor, who will be pleased to consider articles or contributions dealing with modern chemical developments or suggestions bearing upon the advancement of the chemical industry in this country. Communications relating to advertisements or general matters should be addressed to the Manager.

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## 1929:

### A Year of Progress through Unsettled Conditions

As Sir William Alexander, M.P., truly says in his introductory review of British chemical industry in this issue, the year 1929 is 'one in which definite progress may be recorded in several directions, but the progress has been checked and limited by unsettled conditions. The first check to industry came from the General Election, with the unsettlement inevitable to a change of Government and to a natural uncertainty as to the lines of future policy. The second came from financial collapses, both in this country and in the United States, on a scale without precedent. The effects of the losses that have been experienced penetrate far, and some time must pass before they entirely disappear. Notwithstanding all this, the figures of chemical overseas trade are very much better than those of national trade as a whole. November, as we

pointed out last week, was the best month of the year; for the eleven months of 1929 there has been a large increase in chemical exports, which, though unfortunately accompanied by an even larger advance in chemical imports, is nevertheless very encouraging. For the past few years a persistent attempt to make the claims of British chemical products better known throughout the Empire and in foreign markets has been organised, and the results are now beginning to appear in increased consumption of these products abroad and in the deservedly high reputation for quality that they now enjoy.

In the comprehensive article by Mr. Davidson Pratt, the conditions of British chemical industry in practically all its branches are thoroughly explored. Here, again, there are many points that justify a feeling of satisfaction; at the same time the emphasis lies mainly on the increased effort required in the future if we are to maintain and improve our position in the face of world competition. Mr. Pratt puts the case quite frankly: "We are facing steadily growing competition in practically every market, even inside the Empire. We must find means of maintaining our competitive efficiency in the world market. Better organisation of the industry is a first essential, but there are political considerations which may have an even greater influence. We have to compete against countries which, with their home trade secure behind their tariff walls, can send their surplus production abroad at cut prices, with which this country with her largely unprotected home markets is in many cases unable to compete. Unless some solution of this problem can be found the future will be difficult indeed."

Amid all the changes and developments one need remains constant—that of sustained and ever-increasing attention to research. Compared with the pre-war situation, enormous strides have been made in this matter, not merely by private enterprises, but collectively through the research organisations associated with many industries and directed with judgment and sympathy by the Department of Scientific and Industrial Research. We are glad to be able to publish so informed and authoritative an article as that on another page on the work of this Department, in connection not only with the investigations at the various research stations but with specific researches of a fundamental character likely to produce results of general interest and importance. The field covered is already a wide one, including work on the corrosion of metals, synthetic resins, low-temperature tars, hydrogenation of coal, high-pressure gas reactions, water softening, chemotherapy, free lime determinations in Portland cement, the freezing of colloids, and chemical work on fruit.

Synthetic nitrogen products, an entirely post-war development in this country, now represent one of

our largest and most important industries. The year's scientific progress in this field is reviewed by Dr. E. B. Maxted with his accustomed thoroughness and knowledge, and the special developments in the fertiliser branch of the industry are described in a separate contribution. In the latter some account is given of the romance of the Billingham enterprise, with notes on the latest two forms of synthetic fertiliser produced in this country—known as Nitro-Chalk and Ammonium Phosphate. Dr. Maxted points out that the general position in the nitrogen industry continues to contain a curious anomaly, in that, while the world production is still slightly in excess of consumption, most of the important synthetic ammonia companies continue to increase their manufacturing capacity. This probably merely represents a long-sighted manner of dealing with the undoubtedly rising demand for nitrogen, and with the future increase in consumption which is to be expected as a result of educational propaganda. "It is," as he remarks, "impossible to calculate, even approximately, the probable saturation capacity of the world market for fixed nitrogen, but it would appear that this has undoubtedly not yet been reached; perhaps it is not too much to say that saturation of potential demands has not yet been approached."

The British dyestuffs industry—another notable post-war achievement—has long since passed the experimental stage, and ranks among the accepted industries of the country—small perhaps in size and financial turnover, but vitally important as a national nursery of organic chemistry, as the nucleus of a school of organic chemists, and as an essential reserve in national emergency. Mr. L. J. Hooley writes, as in past years, on the scientific and technical aspects of the industry, which reveal steady progress without any sensational new discovery, while the commercial side of the industry is reviewed especially from the consumer's point of view. To the user, the price factor is obviously of first-rate importance, and by degrees the industry is meeting the demand for reductions that bring the British prices nearer to world competitive figures. The colour users are now engaged on the preparation of a report for submission to the Development Committee, who have to present a complete report for the information of the Government, the trade, and the public. The recommendations in this report will have an important bearing on any action to be taken when the original period expires for which the Dyestuffs Act was passed. In the meantime the demand persists for fuller statistics of dyestuffs production, etc., of the kind that the United States Government has long issued. At the moment a dyestuffs patent action is proceeding in the High Court between I.C.I. and the I.G., the results of which are awaited with considerable interest.

Among other features of the present issue attention may be drawn to Mr. J. Arthur Reavell's valuable collection of facts concerning what British chemical plant manufacturers and makers of raw materials for the industry have accomplished during the past year; to Mr. Parrish's discerning discussion of conditions in the heavy chemical branch; to our Patents Correspondent's scholarly résumé of the chief chemical inventions of 1929, in the course of which attention

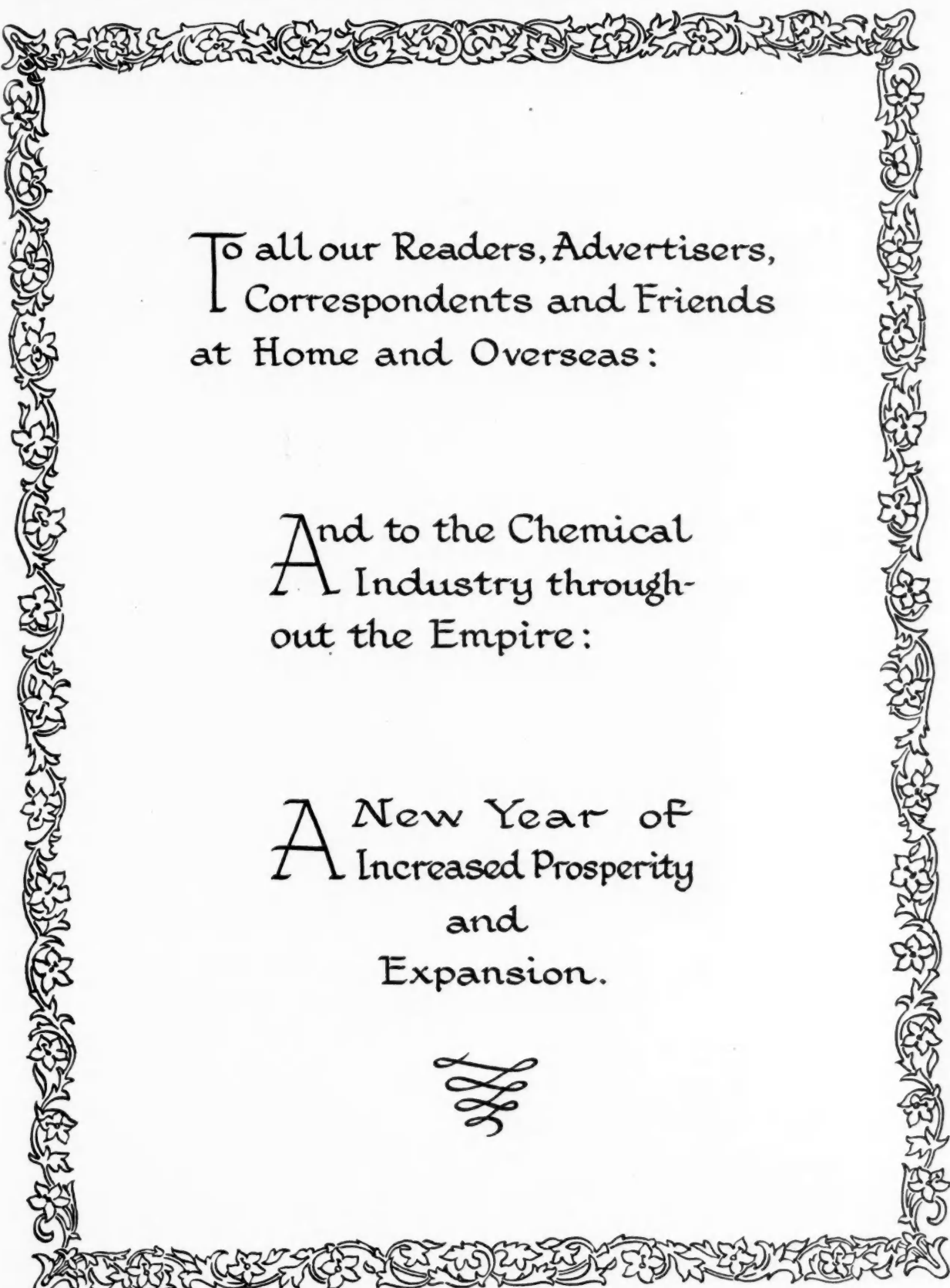
is again drawn to the great preponderance of German inventions patented in this country; and to the article contributed by an eminent authority in close contact with the solvents industry, in which the latest advances in this increasingly important branch of applied chemistry are noted.

Finally, not least impressive among the contents of this number is the record of the work of the various chemical and allied societies. These organisations now constitute a very large group, illustrating the remarkable diversity of the industry itself, and, numerous though they be, one cannot specify any that could be dispensed with without some loss. Their number and the variety of their functions make the problem of their consolidation, even on the loosest federal basis, one of great difficulty.

In one closing sentence: 1929 is still in the main a year of progress, in spite of unhelpful circumstances; with better conditions, one may reasonably hope that 1930 will improve on the present record.

### The Calendar

Jan. 6	Society of Chemical Industry (London Section): "The Micelle Chemistry of Cellulose." C. J. J. Fox. "Meta-filtration." J. A. Pickard. 8 p.m.	Burlington House, Piccadilly, London.
8	Institute of Fuel: "Fuel Economisers with special reference to their Construction, Materials and Recent Developments." O. Kubalek.	Burlington House, Piccadilly, London.
9	Institute of Metals (London Section): "The Aluminium Industry." G. Mortimer. 7.30 p.m.	83, Pall Mall, London.
9	Society of Chemical Industry (Bristol Section): "Wood Distillation." F. G. Conyers. 7.30 p.m.	University, Bristol.
9	Institute of Chemistry (Manchester Section): "The Medical Witness." Dr. R. M. Brontë.	Manchester.
10	Chemical Engineering Group: "Autogenous Welding in Chemical Works." J. R. Boor. 8 p.m.	Burlington House, London.
10	Oil and Colour Chemists' Association (Manchester Section): "Recent Research on Fats bearing upon the Drying of Oils in Paint and Varnish." Professor T. P. Hilditch. 7 p.m.	Milton Hall, Deansgate, Manchester.
10	Society of Chemical Industry (Manchester Section): "Some Aspects of Surface Chemistry and their Industrial Implications." Dr. E. K. Rideal. 7 p.m.	17, Albert Square, Manchester.
13	Institute of Metals (Scottish Section): "Extrusion, and a Consideration of some of the Physical Properties Affecting the Production of Rods, Tubes, and Sections by this Process." A. Wragg. 7.30 p.m.	39, Elmbank Crescent, Glasgow.
14	Institute of Metals (N.E. Coast Section): "Chromium Plating." L. Wright. 7.30 p.m.	Armstrong College, Newcastle-on-Tyne.
14	Institution of Petroleum Technologists: 5.30 p.m.	John Street, Adelphi, London.
15	Society of Glass Technology.	Sunderland.
15	Institute of Chemistry (London Section): Visit to Laboratories and Bakeries of J. Lyons and Co.	London.
15	Society of Dyers and Colourists (Midlands Section): "Measurement of Fading." Dr. S. G. Barker.	University College, Nottingham.
15	Society of Chemical Industry (Newcastle Section): "Methods of Filtration in Industry." R. D. Burn. 7.30 p.m.	Armstrong College, Newcastle.
16	Chemical Society. 8 p.m.	Burlington House, Piccadilly, London.



To all our Readers, Advertisers,  
Correspondents and Friends  
at Home and Overseas:

And to the Chemical  
Industry through-  
out the Empire:

A New Year of  
Increased Prosperity  
and  
Expansion.



## British Chemical Industry in 1929

By Brig.-Gen. Sir William Alexander, K.B.E., C.B., C.M.G., D.S.O., M.P.

*The distinguished writer of this introductory survey of British Chemical Industry during 1929, who is almost equally well known in connection with chemical and allied industries in Great Britain and the United States, deals frankly with the unsettling influences that have hampered progress during the year, but indicates many satisfactory points in the situation.*

AN examination of the reviews contributed under the above heading during the past few years shows that, while details have varied of necessity, and while some of the hopes expressed at the end of one year have been frustrated by the unexpected behaviour of its successor, yet certainty of outlook has been maintained and certainty of progress recorded in one very important direction.

For some years now these reviews have stressed the fundamental importance of research on new products and methods, leading to the establishment of scientific technique for all branches of chemical industry.

The impetus towards what may fairly be called a radical change of outlook and method was one of the products of the late war years. Chemists and physicists have ceased to be luxuries in industry, and are now the essential craftsmen who lay the scientific foundations on which the superstructure of successful industry and commerce is raised.

In last year's review I stated that if normal conditions continued, 1929 and succeeding years would be distinguished by an application of science to industry in all its branches with a skill and intensity hitherto unattained.

Conditions were normal for the first half of the year. Our trade improved at home and was good generally on the Continent—particularly in Germany and the United States of America.

The progress referred to above, so far as home industries are concerned, can be instanced by such examples as the bulk production of synthetic acetone, acetic acid and butyl alcohol by British Industrial Solvents, Ltd. (an associate of the Distillers Company, Ltd.) and of synthetic methyl alcohol, nitric and hydrochloric acids by Imperial Chemical Industries, Ltd., at Billingham.

Substantial progress has also been made in the increased production of sulphate of ammonia and of fertilisers generally, new plants being now in steady and successful operation.

### A Word of Warning

This growth in the adoption of synthetic methods of manufacture, distinguished by close scientific control at all stages, is leading to a situation deserving of analysis and calling perhaps for a word of warning.

The older methods of chemical manufacture, such as the Leblanc process for carbonate of soda, the Chamber process for sulphuric acid, the processes for the manufacture of such products as soap, ultramarine, etc., whilst based on science, were to some extent empirical and dependent for their success on the personal experience of individuals.

Because of long experience and knowledge of the "art," some manufacturers were more successful than others. This kind of success, perpetuated by the handing down of traditional methods and manipulative secrets, is becoming much rarer. The newer synthetic methods, emphasising the purity of reagents and of catalysts and aiming more and more at the development of a fool-proof technique, are creating industries in which manipulation is simplified and operations controlled by careful observations of instruments, thus becoming less dependent on personal technique once the methods are scientifically worked out.

Secrets are more difficult to keep, and if money be available, plants which in many cases are comparatively moderate in capital cost in relation to the older plants can be erected anywhere and worked successfully.

One need but mention the world development of the synthetic nitrogen products industries. There is a possibility of production going ahead of consumption, and there appears, for example, to be to-day an excess productive capacity for sulphate of ammonia.

Whilst competition is good for the consumer, there is a danger actually of uneconomical competition for world trade between nations equipped with all the latest processes.

### Unsettling Influences

To return to the present year, the progressive trade of the earlier months, assisted by a closer and more intelligent co-operation between Capital and Labour, was interfered with by the General Election and by a wave of unjustified speculation in industrial shares, both at home and in the United States of America.

A change of Government must always create a feeling of uncertainty as changes of policy are inevitable. The period of uncertainty is more or less prolonged, dependent on the nature of the new policy and the clearness with which it is defined. It would be idle to deny that the present policy foreshadows a necessary increase in taxation, direct and indirect. Advanced ideas of social reform and of industrial control, together with the possibilities of changes in fiscal and import duties, have combined to create an atmosphere unhelpful to trade recovery and development at the present time.

Financially, there has been a very serious disturbance of the conditions favourable to the progress of chemical industry. There have been many companies started to exploit untried inventions, capital being asked for and obtained in some cases on nothing more solid than an application for a patent. Unduly optimistic prospectuses have led the public to rash investment followed by speculation in shares, leading to loss not only of wealth but of credit and faith.

The recent boom and collapse in the United States of America have been an object lesson to the world, and their results on industry have been unsettling. "Tragic," even, would not be too strong a word, as all this has happened at a time when trade was good and improving. It might be compared to the wilful inoculation of a disease germ into a healthy organism.

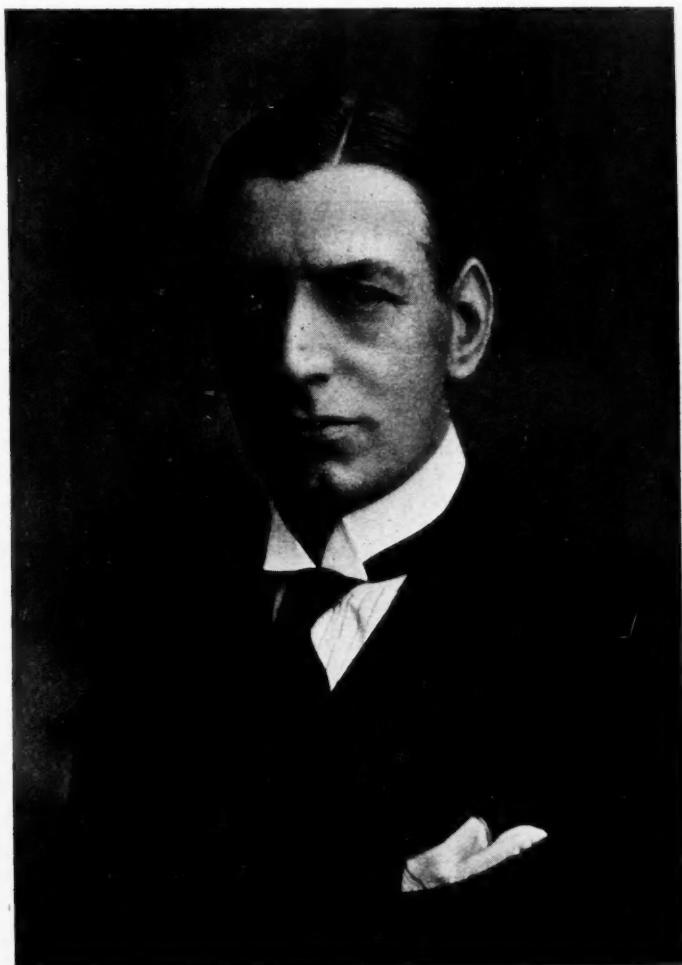
If, however, the collapse has acted as a purge and if the lessons have been learned, then the organism will recover its health, and quite possibly, quickly.

It is important to note the action taken by the United States of America to restore confidence and to stimulate industry. It has decided upon a £30,000,000 reduction in taxation. The prospect of any reduction for us at home is remote and vanishing.

### Essential Conditions of Progress

The picture is a sombre one, and is not lightened by a consideration of what has happened in the great cotton and wool industries. The conditions in the Lancashire cotton trade have not been so bad for many years and the whole industry still awaits a long overdue reorganisation and rationalisation.

Whilst all the basic industries are consumers of chemicals in one form or another, industries in which the new synthetic methods of production have been adopted are relatively



BRIG.-GEN. SIR WILLIAM ALEXANDER, M.P., etc.,  
*a well-known figure in British and International Chemical Industry,  
who contributes a review of "British Chemical Industry in 1929."*

much larger consumers of chemicals. As these new industries prosper, their direct and indirect influence upon the chemical trade will continue to be more pronounced, but the rate of development, so far as this country is concerned, will be controlled by a number of all-important factors, which are not sufficiently realised to-day by the vast majority of our population, and especially by politicians endeavouring to lay down principles without expert knowledge of the subject under discussion.

In order to compete in the markets of the world, production costs must be on a parity with those of competitors abroad. Selling and distributing organisations must be efficient to ensure a minimum of distribution costs. Capital must be available for the amortisation of obsolescent plants, and methods found for drastic reduction in over-head and standing charges in factories, all of which are elements in the cost of production and must directly and indirectly

affect adversely the amount available for the wage item where intense international competition fixes the selling price.

The industries of this country to-day, in comparison with our greatest overseas competitors, are staggering under an abnormal load of direct and indirect taxation which is quite unjustified and quite unnecessary under proper and sane economical administration and policy.

These observations do not make cheerful reading, but they are felt to be called for by the conditions of to-day, which are in part the product of the past year.

Nevertheless, in spite of burdens and uncertainties, there is comfort to be found in the steady growth of the newer chemical industries, in their capacity for meeting new demands and fresh needs, and not least in the movement towards concerted effort in all branches of the industry and in the determination to face realities.

## Recent Progress in the Gas Industry

By Sir David Milne-Watson, LL.D., D.L.

*The contribution of the following article by an authority who might be described as the head of the British Gas Industry indicates how far gas activities now come within the scope of "chemical industry." It discusses the carbonisation and production figures for the industry, experimental work on low-temperature processes, the reduced value of certain gasworks by-products, and other interesting features and problems.*

THE early part of 1929 will long be remembered by the managements of gas undertakings as a period during which the productive capacity of their plants was put to a severe test, owing to the unprecedented demand for gas during the cold weather. The production of gas during the previous year had shown a slight increase in spite of an unusually fine summer, and there is no doubt that when the statistics are compiled at the close of the present year it will be found that the industry is in a healthy and progressive condition.

It is interesting to note that although the quantity of gas manufactured during recent years has shown a substantial and fairly steady increase there has not been a correspondingly large increase in the quantity of coal carbonised. The main increase in the output of gas has been from coal carbonising plants rather than water gas plants, and thus it is clear that, taking the country as a whole, the quantity of gas produced in retorts per ton of coal is still increasing.

Year.	Total coal used in tons.	Cubic feet of gas per ton of coal.*
1922 .....	16,556,134	12,696
1923 .....	17,202,476	13,229
1924 .....	18,104,400	13,381
1925 .....	17,798,725	13,584
1926 .....	17,319,274	13,678
1927 .....	18,456,293	13,902
1928 .....	18,317,969	14,226

\* The figures in this column relate to "Authorised" undertakings only and are exclusive of gas made in water gas plants.

The results shown in the above table may be due in part to the greater use of steam during the carbonisation process, but there is no doubt that they are also the result of steadily increasing technical efficiency.

### The Coal Question

In passing it may perhaps be suggested that although finality in these developments of carbonisation has by no means been reached, the rate of increase in the ratio of gas to coal is not likely to be maintained. If this is accepted it follows that the future increases in the demand for gas which may be anticipated with confidence must result in a substantial increase in the demand for coal by the gas industry provided that we are able to rely upon regular supplies of coal of the required quality. If, however, there is any serious disturbance of supplies it is likely that, as during and after past disturbances, the increased

demand will be met by an increase in the manufacture of water gas.

Any increase in the quantity of coal carbonised will be of benefit not only to the coal mining industry but to the community as a whole, since in the gas and coke oven industries coal is not merely a fuel to be burnt but the raw material of a series of chemical processes.

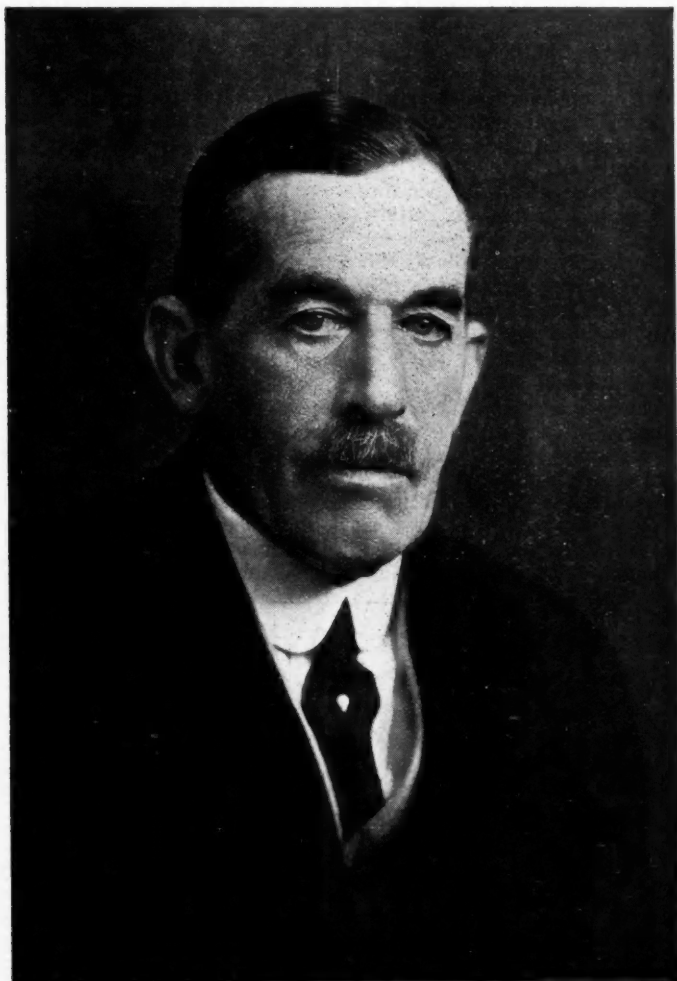
### Coke Ovens

At present about 6,000,000,000 cu. ft. of the gas distributed for general towns purposes is purchased from coke oven plants. The possibility of a further linking up of coke ovens and gas works within a limited area in the Midlands is under active investigation by the Area Gas Supply Committee. Meanwhile it is of interest to note that the Gas Light and Coke Company has recently placed an order for the erection at Beckton of a coke oven plant having a capacity of 1,200 tons per day. This plant will include the usual gas purification and by-product recovery apparatus, and the site on which it is being laid down will readily permit of an extension up to 4,800 tons of coal per day if it is found in practice that this system of gas making satisfactorily meets the requirements of an undertaking whose primary responsibility is to maintain an unfailing supply of gas of uniform heating value at the lowest possible price.

Several systems of low-temperature carbonisation are under trial in different gas works, and four of these in London were recently inspected by members of the Institute of Fuel. It is significant that discussions of this matter are no longer concerned with technical details, but resolve themselves into a demand for a statement of the economic results of commercial working. Nowhere has this demand been more persistent than in the gas industry, and now it appears that results as to technical efficiency, capital and labour charges, ground space requirements and value of by-products should be available in the near future.

### Low-Temperature Tar

The outstanding technical uncertainty of low-temperature carbonisation is the utilisation of the tar in quantities such as would be produced if any considerable tonnage of coal was carbonised in this way. This difficulty of disposal at remunerative prices has been intensified within the last eighteen months by the downward movement of creosote prices in Europe, following the extension of tar distillation



SIR DAVID MILNE-WATSON, LL.D., D.L.

*Governor of the Gas Light and Coke Co., who reviews "Recent Progress in the Gas Industry."*

plants in America. The inventive chemist might well note that creosote can be obtained in large quantities to-day at prices considerably below those which have prevailed for some years past.

These developments in connection with coke ovens and low-temperature carbonisation plants are evidence that the methods of gas making are still to some extent in a state of flux. This is a healthy sign that no opportunity of future improvement will be missed. Rather may we anticipate that as each unit of plant wears out it will be replaced not necessarily by a replica, but by what at the moment appears to be most efficient apparatus having regard to local conditions. Thus at the present time new installations of horizontal retorts, continuously operated vertical retorts, intermittently charged vertical chambers, complete gasification plants and water gas plants are under test in addition to the coke ovens and low-temperature carbonisation plants already mentioned.

In connection with tar generally and with low-temperature tar in particular, there is considerable research activity in various parts of Europe with the object of converting products of high boiling point into spirit suitable for the internal combustion engine. These processes have hardly emerged from the experimental scale, but several which involve the hydrogenation of tar under pressure appear to be worthy of the closest consideration.

#### Extraction of Benzole

There is an increasing tendency to extract benzole from coal gas for use as a motor spirit. In the past the plants adopted for this purpose have involved the washing of the gas with some form of oil. A new plant in which activated charcoal is used as the absorbent for benzole has just completed twelve months successful working in this country. Several such plants have been working for longer periods on the Continent and it is claimed that

the crude benzole recovered may be used without the extensive rectification which is necessary in the case of benzole recovered by oil washing.

The reduced value of by-product ammonia is still a matter of serious concern to gas undertakings, indeed in some cases the disposal of ammoniacal liquor represents a charge upon the manufacture of gas. Research is being actively pursued in several directions, but it is clear that the time has not yet come when the industry can look for any heroic or spectacular solution of this problem. Much may be achieved, however, by the patient application of simple chemical engineering principles in many of the older gas condensation and ammonia washing plants.

The practice of the partial dehydration of gas prior to its distribution to the public is extending and may possibly be stimulated in the future by recent legislation which has rectified anomalies in the testing of such gas by public authorities.

#### A Large Field for Gas

It has been stated above that further increases in the demand for gas may be anticipated. It should be realised, however, that a well-informed direction of the selling organisation will be necessary to ensure this. The increased business of recent years has been largely secured by the more general installation of what may be called mass production apparatus. In certain districts it is possible that further extension of business must depend upon the development of specialised forms of apparatus, the design of which will demand the detailed attention of the technician with appreciation of the principles of combustion and gas flow. There is no doubt, however, that in many industries, as well as in hotels, shops, offices and modern blocks of flats there is a very large field for the further extension of special designs of gas-fired apparatus for many purposes.

## British Chemical Industry: A Review of the Past Year

By J. Davidson Pratt, M.A., B.Sc., F.I.C.

(General Manager of the Association of British Chemical Manufacturers.)

*For a second year we are indebted to the author for an exceptionally comprehensive and authoritative survey of the whole field of British Chemical Industry, as seen from the point of view of chemical manufacturers. While there is in various directions much cause for satisfaction, Mr. Pratt reminds our readers of several problems that need to be faced frankly.*

PRODUCTION statistics for the chemical industry as a whole do not exist, so any opinion as to the progress made must be based on the views of the main manufacturing concerns and on such figures as are published by the Board of Trade in regard to indices of production, imports and exports. Taking 1924 as 100, the index of production for 1928 for the chemical and allied industries, as defined by the Board of Trade, was 110.3, which was somewhat higher than was anticipated. The figures for the first three quarters of 1929 have been 107.2, 112.9, and 117.5 respectively, thus showing a gratifying rise in spite of the continued depression in many of the chemical consuming industries, such as textiles, etc. The last quarter's figure shows a remarkable advance since the corresponding period last year, when it was 106.3.

On the other hand, the unemployment figures are not so satisfactory. The percentage of insured persons unemployed in the chemical industry, as defined by the Ministry of Labour, was 5.9 in October 1928; it rose to 6.9 at the end of January 1929, fell in April to 6.0 and has, since August, been in the neighbourhood of 6.5.

The import and export figures provide food for serious reflection. The imports of chemicals, drugs, dyes, and colours for 1928 were close on £15,500,000, and showed a slight reduction on 1927. For the first ten months of this year these imports have already exceeded £14,000,000, and are more than 10 per cent. higher than for the corresponding

period in 1928. The exports for 1928 nearly reached £25,500,000, which was about £2,000,000 higher than in 1927. This upward tendency has not been maintained; several times the monthly exports have dropped below the 1928 level, but for the first ten months of this year the total shows a reassuring increase of about £400,000 as compared with 1928.

These figures are significant, and indicate the growing intensity of foreign competition and an increase in some directions of what can only be called "dumping," against which in the fine chemical field even the 33½ per cent. duty under the Safeguarding of Industries Act does not afford adequate protection.

#### Heavy Chemicals

Speaking generally, the home trade in heavy chemicals has shown a steady improvement in spite of adverse influences such as the unrest in the cotton and woollen industries, the continued depression, now showing signs of lifting, in the steel trade, and a temporary set-back in the production of artificial silk, which has not made such a big demand on heavy chemicals as last year.

As regards prices, there has been a steady decline for the past few years; this cannot be expected to continue, and in most directions rock bottom, from the economic point of view, has been reached. In some cases prices have actually started on the up grade. If the reorganisation in the coal industry has to mean higher prices for home trade,

then an increase in the cost of chemicals must also be faced. The new burden of social services may also prove another adverse factor, unless some means can be found of providing the necessary funds without resorting to increased taxation.

As regards foreign trade, increased exports are recorded for the first ten months of 1929, as compared with the same period in 1928, in respect of sulphuric acid, sodium carbonate (including soda ash, crystals and bicarbonate), sodium sulphate (including salt cake), zinc oxide and miscellaneous chemicals, while there have been decreases for tartaric acid, ammonium chloride, bleaching powder, copper sulphate, glycerine, potassium compounds, caustic soda and miscellaneous sodium compounds; the net decrease in the heavy chemical export trade is approximately £250,000. These observations do not take account of ammonium sulphate or coal tar products, which are dealt with later. The imports are not very instructive, as the bulk of the increase is in the case of miscellaneous unspecified compounds. Increases occur in respect of all the chemicals in the import list, the most noteworthy being acetic acid, tartaric acid, bleaching materials, calcium carbide and potash salts.

#### Sulphuric Acid.

As regards the sulphuric acid industry, it is satisfactory to be able to report an increase in the proportion of plant capacity in operation. During 1928 this figure was 65.8 per cent., whereas during 1929 the figure was 70.7 per cent., an increase of 7.5 per cent. on last year's figure. In terms of tonnage of sulphuric acid (as 100 per cent.  $H_2SO_4$ ) the amount produced in the United Kingdom and Ireland during 1928 was 908,000 tons, whereas during 1929 it has increased to 966,700 tons, this being the highest figure recorded for some years. (In this case the years end on September 30.)

Some interesting comparisons of data during recent years are contained in the following tables. It should be noted, however, that the figures refer in these tables to the year ending December 31:—

TABLE I.

Year	Acid made, including Oleum, in tons of 100% $H_2SO_4$	Percentage Made from					Zinc Concentrates
		Pyrites	Spent Oxide	Brimstone			
1924	918,000	48.5	1.0	22.8	23.6	4.1	
1925	848,000	45.94	1.1	23.82	23.67	5.57	
1926	722,000	46.2	0.92	24.57	23.18	5.13	
1927	889,000	42.37	1.16	24.44	24.6	7.42	
1928	928,000	44.18	0.81	25.64	20.12	9.25	
1929 (6 months of)	488,000	47.93	1.01	24.23	17.56	9.27	

TABLE II.

Production of Sulphuric Acid and Consumption of Raw Materials in Tons. 100 per cent.  $H_2SO_4$

Year	Acid and Oleum	Net Acid	Oleum	Pyrites	Spent Oxide	Sulphur & $H_2S$	Zinc Ores
1924	918,000	874,000	44,000	360,000	152,000	69,700	37,100
1925	848,000	779,000	69,000	313,000	148,000	64,200	57,700
1926	722,000	643,000	79,000	268,000	128,600	53,600	51,300
1927	889,000	773,400	115,400	305,500	155,500	68,700	86,300
1928	928,000	782,000	146,000	329,000	174,000	58,300	116,400
1929 (6 months of)	488,000	413,000	75,000	185,000	87,000	26,300	58,300

It will be noticed from these figures that the proportion of the acid manufactured from pyrites has increased, whereas the proportion manufactured from brimstone has decreased, and that the figures relating to spent oxide and zinc concentrates remain substantially constant.

It is worthy of note that the demand for spent oxide has

increased in proportion to the decrease in the use of brimstone, and this has caused a tendency for prices to harden all over the United Kingdom, without, of course, in any way increasing the supplies, and must have the inevitable result of a similar hardening in the price of sulphuric acid.

New developments of importance include the manufacture of acetone from alcohol by a new process, of nitric acid by the oxidation of synthetic ammonia on an extended scale, and of methyl alcohol from its elements, which will render this country independent of overseas supplies of these materials; the production of acetic acid by synthetic means will shortly be a *fait accompli*.

#### Fertilisers

The subject of fertilisers may be briefly dismissed, since it will be discussed in another article. Attention is directed to the recently published annual report of the British Sulphate of Ammonia Federation, which has already been summarised in this journal, and which gives an admirable review of the position as regards nitrogenous fertilisers. Two features need special comment; first, the further reduction of about 10 per cent. in the price of all forms of nitrogen for 1929-1930, which will be an undoubted boon to the agricultural community; secondly, the tendency for production to outrun consumption.

In the latter connection, mention should be made of what may be regarded as an attempt to rationalise the nitrogen production industry on an international basis, namely the agreement which was concluded in the summer between Imperial Chemical Industries, Ltd., the German I.G., and the Chikan Government with a view to the avoidance of a ruinous price-war between natural and synthetic nitrogen, which would not have been in the general interest. Co-operation between these

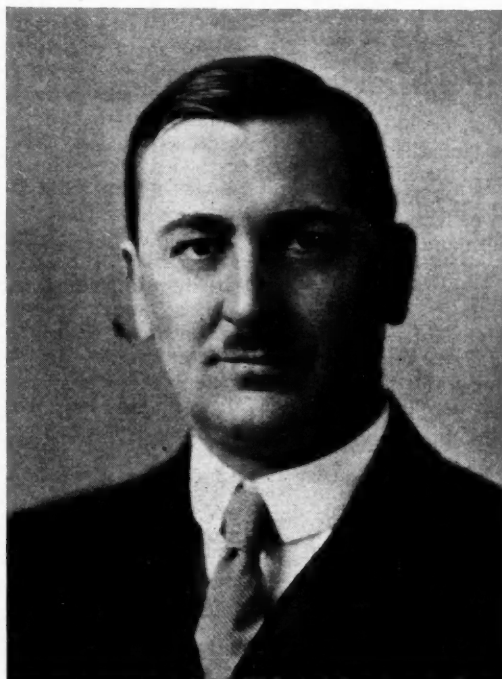
bodies in the production and marketing of nitrogen products and in propaganda is the main feature of the agreement; there is thus an apparent admission that natural and synthetic nitrogenous fertilisers must run hand in hand and neither can be regarded as a complete alternative to the other. The danger is not yet over, and new outlets for nitrogenous fertilisers must be found to absorb the steadily increasing production.

#### An Interesting Imperial Possibility.

In this connection there is a splendid opportunity for rationalisation within the Empire, whereby this country can supply the Colonies and Dominions with the fertilisers they need in return for their wheat and other foodstuffs. This would be an important step towards the consolidation of the Empire and would be to the advantage of all concerned, since it would ensure an outlet for our rapidly developing synthetic ammonia industry and a market for the products of the Dominions concerned.

The exports for sulphate of ammonia to the end of October, 1929, show a gratifying increase of about £900,000 in value over the same period in 1928, Japan being responsible for nearly half of this amount.

The outlook in the superphosphate industry has become more hopeful in the sense that it would appear that the



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imports due to unrestricted foreign competition have reached a maximum. Prices have also improved slightly, while consumption has increased, but the future is not without cause for anxiety.

#### Coal Tar Products

Of general commercial conditions in the coal tar industry there is little to report which is not already too well known. Markets throughout the year have not been as active as during 1928 owing to sub-normal general trade conditions. Pitch and creosote were most concerned, with disastrous effects on prices. In the case of the former, both home and foreign demand was substantially reduced, while the steady decline in the United States demand for creosote was not arrested during the past year. Light fractions were brighter, and while the battle is a hard one, refined tar has brightened up generally. The home demand for anthracene and naphthalene has also been better. The chief feature during the latter part of the year was, of course, the prolonged scarcity of crude carbolic acid and of the crystals. At certain times this amounted to a potential famine, and even resulted in a substantial import into the country.

With regard to foreign trade, the position is very quieting, as the exports to the end of October are about 30 per cent. lower than in the same period last year. All products, with the exception of naphthalene, which has gone abroad in increased quantities, have been affected.

The year has seen the completion of further amalgamations, notably those in the Lancashire and Scottish areas. In the former, the tar distilling interests of North-Western Co-operative Tar Distillers, Ltd., Hardman and Holden, Ltd., Thomas Horrocks and Sons, Ltd., J. E. C. Lord (Bootle), Ltd., and J. E. C. Lord (Manchester), Ltd., were absorbed to form Lancashire Tar Distillers, Ltd.

The Scottish Tar Distillers, Ltd., was formed to take over the tar distillation activities of James Ross and Co. (Lime Wharf), Ltd., and Henry Ellison (Glasgow), Ltd.

Co-operative tar distillers and older amalgamations have in one or two cases extended their scope by the inclusion of hitherto isolated tar distilling firms.

During the year the Bussey plant at Glenboig, Glasgow, came into operation. The set is stated to be the largest low-temperature distillation plant in the world and is the outcome of successful working on two similar but smaller units in the States. Throughput is between five and six hundred tons of coal per diem, and 15,000 gallons of crude oil will be produced from this throughput.

#### Hydrogenation of Tar

There has, from the first reports of German hydrogenation of coal, been much comment on the extent of these activities. Without engaging in an extended investigation on the practice in Germany, the suggestions that have been made during the year, to the effect that the I.G. were not hydrogenating coal but low-temperature tar, deserve mention here, as such a report, whether ill or well founded, is receiving attention by research workers on the Bergius and other systems in this country, and the practical consequences should be watched and indeed investigated by the low-temperature works in this country.

The Tar Boards set up by the British Road Tar Association in different parts of the country have been an undoubted success in developing interest in the use of British refined tar in road making, and well organised and intensive propaganda in this and other ways is bringing credit to that association and increased demand for tar from the tar distillers. This is of the utmost importance in connection with the Government's new schemes for the improvement of our roads.

The work of the Fuel Research Board on the nature and properties of low-temperature tars should not be overlooked. Besides work on the analysis of these tars, it has been possible to determine very closely the factors governing

the relative proportions of the various constituents and the best methods for treatment of a low-temperature tar to effect subsequent adjustment of those proportions.

Nor should the work on free carbon, carried out by the Gas Light and Coke Co., be ignored. Though it may be regarded as of a specialised nature, the research stands as an authoritative investigation of the relationship of free carbon to the history of the original tar and to the methods of distillation, and in those aspects it becomes a work of reference on which future manipulation of gas works and tar distilling practice may be adjusted as market conditions for coal tar products vary.

Work in the laboratories of the British Dyestuffs Corporation on potentiometric and conductivity methods of analysis is also of importance, as from the subsequent use of the data obtained methods have been developed for the examination of coal tar products.

#### Wood Distillation

The wood distillation industry is passing through what is probably the most critical period of its long history. The products upon which the industry chiefly relied, namely, acetic acid, methyl alcohol (wood spirit) and acetone, are now either being produced by new processes at home or are imported at prices with which the wood distillation industry cannot economically compete.

Production costs, owing chiefly to the rates of wages which have to be paid in this country for similar work, are higher than in foreign countries. Competition is rendered still more difficult on account of the higher prices demanded in this country for waste wood, and also the higher rates of transport, both at home and for export, than those obtaining in foreign countries.

Several old-established wood distillation firms have for these reasons been forced into liquidation, and it would appear that only those larger concerns will survive which have vast quantities of waste wood to dispose of and are compelled to distil it, in some cases, even at a loss.

Acetic acid from wood distillation is still finding a ready market, but, as in the case of wood spirit, the price has had to be reduced, in order to meet the competition from new methods of production, to a level which, in many cases, leaves no margin of profit.

The wood distillation industry is further hampered by the importation of foreign wood charcoal, offered at prices which in many cases are below the cost of production in this country, and in consequence some wood distillers are being compelled to act as merchants in order to retain their business connections with this commodity.

In view of this state of affairs, the more important wood distillers are testing new processes which promise to lessen the cost of production—e.g., the Suida method for producing 98 per cent. acetic acid directly from the crude pyrolygneous distillate. The production of active carbon, in the manufacture of which one of the chief products of wood distillation, namely, calcium acetate, is used as the activating agent, is also being explored, the subsequent process of distillation and activation producing acetone from the calcium acetate, and also pyrolygneous acid to make a sufficient quantity of calcium acetate to treat a further batch of wood. This development depends for its success on a much increased industrial use of active carbon, the utilisation of which has until recently not received the attention which it merits on account of its valuable absorptive properties for organic vapours.

#### Dyestuffs

The dyestuffs section of the industry has continued its steady progress, and in spite of depressed conditions in the textile trade, the dye production for 1929 gives promise of exceeding that in 1928, in which the output was the

highest ever attained in this country. The 1928 production was slightly over 22,700 tons as compared with 18,000 tons in 1927 and just over 4,000 tons in 1913. A specially gratifying feature is that the output of dyes belonging to the fastest and most expensive series showed in 1928 an increase of more than 20 per cent. over 1927, the 1928 figure being just under 5,000 tons. The progress made is witnessed by the fact that considerable extensions to works and plant are in course of construction.

#### Increased Exports

The export trade also shows a gratifying increase. So far only the figures to the end of October are available, and these are already in excess of the total exports for 1928, both as regards weight and value, the 1928 figure being 10,200 tons, valued at £905,781. The 1929 totals for the year may, in fact, be expected to show an increase from 20 per cent. to 25 per cent. over 1928, which is most encouraging having regard to the growing intensity of competition in foreign markets.

Imports for 1929 may also be expected to exceed the 1928 figures by about 20 per cent. as regards weight and 10 per cent. as regards value, the 1928 figures being 2,095 tons valued at £1,000,000. Thus at least our exports are likely to exceed our imports in value, though the actual quantity sent out of the country is far larger than that imported, due to the fact that only those specialities which have as yet no counterpart here, are coming in, and consequently high prices are being paid for them by British colour users. This points a useful moral as to the need for the continued development of our dyestuffs industry, if low priced dyes are to be procured.

Substantial price reductions have been effected consequent on a comprehensive review of prices by Imperial Chemical Industries Ltd. at the beginning of the year, and this was followed by a reduction of the price factor from 2 to 1.75 by the Dyestuffs Advisory Licensing Committee in May. Dye users have, therefore, benefited considerably.

Technical development has also proceeded at a satisfactory rate. Among the many new dyestuffs placed on the market by British manufacturers are some of considerable importance in the sense that their advent has filled gaps in the available range which formerly had to be supplemented by importation, under licence, of corresponding products of foreign origin. There are also novelties not hitherto manufactured either in this country or abroad, possessing special features of shade, fastness, or affinity for a particular fibre.

Not only have the energies of the dye makers been directed towards the manufacture of dyewares, but much activity has also been shown by the larger concerns in the production of products related either in process of manufacture, or in chemical constitution, to dyestuffs and intermediates. Interesting contributions to the elucidation of problems connected with the application of dyestuffs to textile and other materials also stand to the credit of both large and small concerns.

#### Fine Chemicals

The trade in fine chemicals has, on the whole, continued its upward trend, but there is no phenomenal feature to report. The year may be described as one of development and consolidation of processes already established, with a consequent tendency for price levels to fall.

In the case of drugs, medicines, and medicinal preparations, the exports for 1928 were valued at £3,000,000. This year for the first ten months there is an increase of 8 per cent. over the same period in 1928. The import position is much less satisfactory. The total imports in 1928 were just over £2,000,000, and this figure has already been exceeded by £70,000, with two months of the present year to go; thus an increase of nearly 30 per cent. in the imports is to be visualised. The exact reasons for this

are not easy to determine, but in many cases there have been undoubtedly imports at declared values much lower than the ruling world price, and against which even the safeguarding duty of 33½ per cent. has been inadequate.

Important developments in the medicinal field have been the extended production and use of Vitamins A and D, of ephedrine for treating asthma, hay fever, etc. and of S.U.P. 36, for the treatment of influenza and like affections. Synthetic menthol is steadily increasing in importance, in spite of a considerable reduction in the price of the natural material, thus indicating that the better class products are destined to become a very serious rival to the natural menthol.

There is nothing special to report regarding laboratory reagents and research chemicals, and the position is satisfactory.

Photographic chemicals have again been in good demand, though not quite to the same extent as last year, probably owing to the dull weather in the early part of this summer. Price levels remain unchanged.

#### Perfumes and Essential Oils

The production of perfumery chemicals has been well maintained, and in a number of lines a very definite increase has taken place. This has been due mainly to a greater home demand, since foreign sales are difficult to effect owing to the very high tariffs in other countries. The level of prices has tended to fall owing to severe competition, and here, again, there are cases in which foreign supplies are being imported in spite of the protective duty, and at prices lower than those ruling in the country of origin.

With essential oils the volume of trade has been maintained, but prices generally have been at a lower level owing in some cases to over-production. There have, however, been a few notable exceptions such as Java and Ceylon citronella oils, oil of limes, Neroli and Bourbon geranium oil, where curtailment of production or failure of the crop has caused a rise in price. Last year's imports of essential oils, excluding turpentine, were valued at £1,200,000, and this year may be expected to be over 10 per cent. higher.

The organic solvent industry has continued its steady progress. Mention has already been made of the new developments in regard to acetone and methyl alcohol. The production capacity for solvents of the cyclohexanol group and for ethyl lactate has been increased so that the requirements of our domestic trade can now be met. The quality of the production compares favourably with that of foreign supplies.

#### Conclusion

While the year's activities may in some directions give cause for satisfaction, the indications for the future are by no means reassuring. We are not securing that share of the foreign trade which is essential for the proper development of the industry, and we are facing steadily growing competition in practically every market, even inside the Empire. We must find means of maintaining our competitive efficiency in the world market. Better organisation of the industry is a first essential, but there are political considerations which may have an even greater influence. We have to compete against countries which, with their home trade secure behind their tariff walls, can send their surplus production abroad at cut prices, with which this country with her largely unprotected home markets is in many cases unable to compete.

Unless some solution of this problem can be found, the future will be difficult indeed. The Empire offers us a wonderful opportunity if it can be consolidated to form a single economic unit; while the difficulties of achieving this are undoubtedly great, the resultant prosperity to all parts of the Empire would be such as to justify the most energetic action towards the attainment of this end.

## The Nitrogen Industry in 1929

By E. B. Maxted, D.Sc., Ph.D., F.I.C.

*The value of Dr. Maxted's annual review of developments in the nitrogen industry has long been recognised by all students of synthetic developments. This is the eleventh article of the kind he has contributed to our Annual Review, and it is as valuable as any of its predecessors.*

THE general position in the nitrogen industry continues to contain a curious anomaly, in that, while the world production is still slightly in excess of consumption, most of the important synthetic ammonia companies continue to increase their manufacturing capacity. This probably merely represents a long-sighted manner of dealing with the undoubtedly rising demand for nitrogen, and with the future increase in consumption which is to be expected as a result of educational propaganda.

The rate of increase of consumption for the past five years has averaged some 160,000 tons of nitrogen per annum; and it has, for the past two years, been at the rate of over 200,000 tons of nitrogen per annum—corresponding with over a million tons of ammonium sulphate, if all the nitrogen were used in this form. Thus, although production may in any year to some degree exceed consumption, new plant, or utilisation of reserve plant, producing roughly 200,000 tons of fixed nitrogen, is necessary each year in order to meet the increased yearly demand; and a relatively large extension may only cover requirements for a few years ahead. It is of course impossible to calculate, even approximately, the probable saturation capacity of the world market for fixed nitrogen; but it would appear that this has undoubtedly not yet been reached; perhaps it is not too much to say that saturation of potential demands has not yet been approached.

### Forms of Fixed Nitrogen

Of the artificially fixed forms of nitrogen, synthetic ammonia continues to hold the paramount position, its production being now considerably greater than by-product sulphate; but there has been during the year a considerable revival in Chilean nitrate production and consumption. For the figures involved, reference may be made to the Annual Report of the British Sulphate of Ammonia Federation (1). In connection with the nitrate position, the discovery of nitrate-bearing lands in South West Africa (2) is certainly of interest; but, until further details of the local facilities for extraction and, particularly, for transport are available, little can be said of the probable importance of these. With heavy chemicals such as nitrate or ammonium sulphate the cost and ease of transport are of cardinal importance, as is evidenced by the special docks which are apparently desirable (3) in connection with the Billingham plant.

While the bulk of the nitrogen continues to be used in the form of ammonium sulphate and sodium nitrate, increasing efforts are being made to produce fertilisers free from inert ballast—namely, without acidic or basic radicals which in themselves possess no fertilising value. Ammonium phosphates and, especially, mixtures containing potassium, nitrogen and phosphorus, in addition, sometimes, to calcium, appear to be the most popular products of this type.

In view of the world-wide use of inorganic nitrogen for agricultural purposes, Professor Fowler's address to the Indian Chemical Society (4) is of special interest. Fowler expresses the opinion that, while mineral nitrogen may be sufficient to cover the main requirements of plants, additional forms of organic nitrogen are required for complete nutrition. The point is one which certainly deserves detailed investigation.

### Synthesis of Ammonia

Interesting details of an ammonia plant at Ostend have been given by F. Pallemerts (5). The hydrogen is manufactured by the low-temperature separation of coke-oven gas and is stated to cost approximately one half of that produced from water gas. The cost of the fixed nitrogen is reported as being 9 cents per kilo. This utilisation of coke-oven gas is one of the newer phases of the synthesis, and is being successfully applied in a number of other plants. Attention may also be called to

a paper (6) dealing with the engineering—and, particularly, the compressing—side of the industry. Compressor design, the problem of cooling, and other points are dealt with in greater detail than is usual in published descriptions of ammonia plant.

A number of fresh specifications covering methods of purifying the gases used for the synthesis have appeared. Thus, the Gasverarbeitungsgesellschaft m. b. H. propose, in order to remove the last traces of water, that the gases should first be passed over absorbents in the usual way, then mixed with a small percentage of ammonia and cooled to a temperature sufficient to cause liquefaction of this ammonia which, in condensing, brings down with it any water vapour that may remain (7). Impure hydrogen, containing methane, may, according to a method (8) suggested by the I.G., be freed from catalyst poisons, including water, by being scrubbed with liquid methane, the latter being readily obtained in the course of fractionation of coke-oven gas at low temperatures. After this washing, any unremoved inhabitants may be adsorbed by silica gel or active carbon.

A new absorbent for purifying the gases has been proposed by C. Müller and F. Krägeloh (9), who recommend treatment with anhydrous potassium aluminium ferrocyanide at a somewhat lower temperature than that used for the actual synthesis. This method resembles somewhat the previous treatment of the gases with catalyst in a preliminary furnace, as was proposed early in the history of the synthesis, since complex ferrocyanides of this type are, as will be seen below, one of the catalysts used for the actual synthesis.

### Catalysts

The use, as a synthesising agent, of a catalyst of the above type has been protected by F. Uhde (10). The potassium ferrocyanide and aluminium chloride are evaporated together and roughly powdered, then reduced with hydrogen at 300–450°C. The catalyst thus formed is sufficiently active to be used at a working temperature of 350–450°C. and possesses a relatively long life. Precautions which are adopted in order to obtain such catalysts in a state of optimum activity are described by Uhde in a further patent (11), according to which catalysts containing complex cyanides are reduced in the reaction vessel itself with a mixture of nitrogen and hydrogen from which all traces of oxygen and its compounds have been removed. The nitrogen may contain, for instance, ten per cent. of hydrogen at first, this being raised, together with the temperature and pressure, as reduction proceeds, until a normal gas mixture and normal conditions of temperature and pressure are obtained.

For use under somewhat similar conditions, namely at relatively low temperatures and pressures, A. Hurter (12) recommends double cyanides of iron and the alkali metals together with the oxide, or certain other compounds, of zirconium. As before, the mixture is evaporated to dryness and reduced with hydrogen at a temperature and pressure which are gradually raised to 400° and 90 atm., respectively. A suitable mixture consists of 10 parts of potassium ferrocyanide, dissolved in 40 parts of water, and 5 parts of zirconium oxychloride in 30 parts of water. Finally, a catalyst consisting of a zeolite, or of non-siliceous base-exchanging bodies generally, together with a catalytically active constituent, has been described by the Selden Company (13).

A somewhat unusual method of activating catalysts for the synthesis of ammonia is put forward by the I.G. (14), the catalyst being exposed to Röntgen radiation before use. It is

(1) THE CHEMICAL AGE, Vol. XXI, p. 465.

(2) THE CHEMICAL AGE, Vol. XXI, p. 102.

(3) THE CHEMICAL AGE, Vol. XX, p. 115.

(4) *Chemistry and Industry*, 1929, p. 673.

(5) *J. Ind. and Eng. Chem.*, 1929, 21, 22.

(6) *Chemistry and Industry*, 1929, p. 591.

(7) Brit. Pat. 293,631.

(8) Brit. Pat. 299,558.

(9) U.S. Pat. 1,727,174.

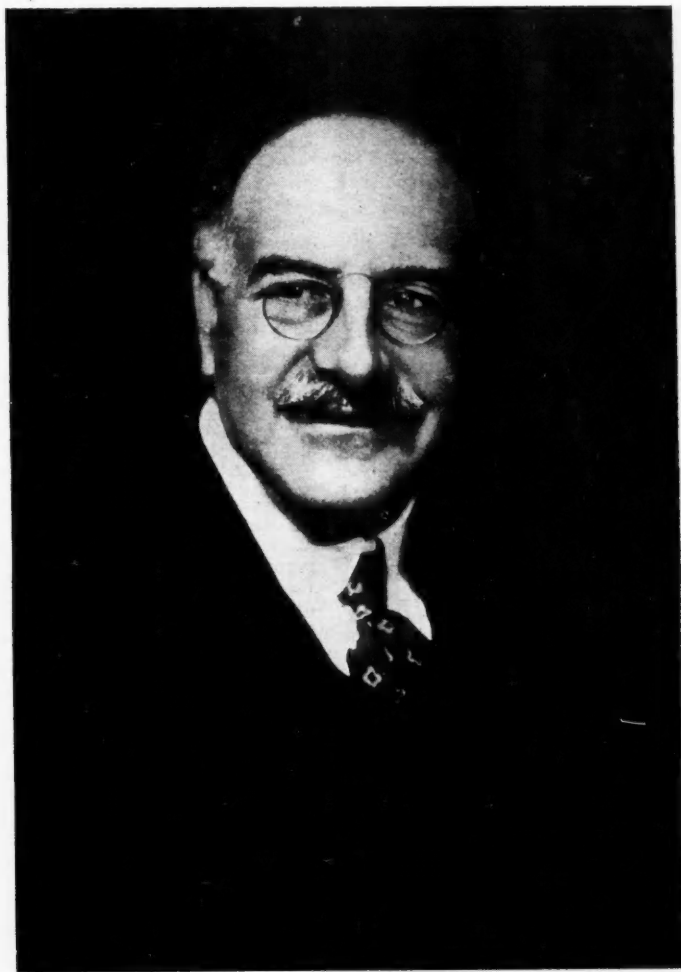
(10) Brit. Pat. 273,735.

(11) Brit. Pat. 272,930.

(12) Brit. Pat. 305,753; *THE CHEMICAL AGE*, Vol. XX, p. 258.

(13) Brit. Pat. 307,457.

(14) Brit. Pat. 315,900; *THE CHEMICAL AGE*, Vol. XXI, p. 177.



THE RIGHT HON. LORD MELCHETT,

*the Chairman, the founder, and the directing mind of the great British and Empire organisation known as Imperial Chemical Industries, Ltd. Eldest son of Dr. Ludwig Mond, he continues with distinction a great family and scientific tradition in the field of industry.*

stated that catalysts treated in this way give higher yields of ammonia than similar, but untreated, catalysts. It may be recalled that the method has been used also for hydrogenation catalysts with, however, varying results.

While a number of types of plant employ a variable temperature throughout the catalyst mass, R. S. Richardson and the Nitrogen Engineering Corporation (15) now propose to pass the gases used in the main circuit of the synthesis first of all through a relatively small mass of catalyst at a relatively high temperature, additional gas being subsequently added in order to cool this to the normal working temperature, when it is passed through the main catalyst chamber, which contains a far larger mass of catalyst than that used for the preliminary treatment.

The possibility of using natural gases for the synthesis of ammonia has been discussed by H. Liander (16), with special reference to mixtures containing methane. For details, the original papers should be consulted. Such a use would, of course, materially cheapen the process at places at which such gases are available.

#### Ammonium Salts

In view of the interest which is attached to ammonium salts containing acid radicals which are themselves active, attention may be drawn to a specification (17) of the I.G., according to which soluble calcium phosphate is treated with an acid, for instance with hydrochloric acid, under conditions such that mono- or di-calcium phosphate is precipitated. This is separated and treated with ammonia or with a mixture of ammonia and carbon dioxide. A cyclic method of converting ammonium chloride into free ammonia and hydrochloric acid has been described by Kessler (18). The vapour of the ammonium chloride is brought into contact with fused ammonium bisulphite, when hydrochloric acid is given off and ammonia is absorbed. This ammonia may be expelled from the mass by means of a current of superheated steam, preferably after any residual hydrochloric acid has previously been expelled by treatment with air.

Slade and Imperial Chemical Industries, Ltd. (19), recommend in the preparation of ammonium sulphate by double decomposition of calcium sulphate and ammonium carbonate, that, after filtering off the calcium carbonate in the usual way, the residue should be heated to drive off ammonia and carbon dioxide. On crystallising, the ammonium carbonate separates before the sulphate. Finally, among the devices for obtaining crystals of ammonium sulphate and other bodies of uniform size, may be mentioned a specification of the I.G., according to which (20) a vertical saturator is used, through which an air current is blown in order to keep smaller crystals in suspension, while those of larger size sink.

#### Nitric Acid and Nitrates

A practical paper dealing with general routine and various technological points in connection with the manufacture of nitric acid from ammonia has been contributed by M. Kaltenbach (21). In the plant described, the ammonia is transported in liquid form, distributed in the usual way and oxidised in converters of the Parsons cylindrical type. The general routine of absorption of nitrogen oxides is also treated. G. A. Perley and M. W. Varrell (22) have compared various arrangements of platinum gauze, including double and multiple gauzes in substantial contact and separated by a definite distance. Little difference in efficiency was observed with any of the arrangements tried; indeed, it would appear that, provided intimate, even if exceedingly short, contact between the platinum and the gas mixture is obtained, any further contact with the gauze, by virtue of the exceedingly high reaction velocity, does not increase the yield, and may depress this, owing to decomposition of nitrogen oxides. Platinum-rhodium alloys are stated by the Du Pont de Nemours Co. and C. W. Davis (23) to be more effective than platinum alone for this reaction and to be more resistant to high temperatures. The alloys suggested contain from 2 to 50 per cent. of rhodium. It is well known

that the activity of platinum can be influenced for other reactions by alloying with a secondary constituent; but the widespread use of pure platinum itself has continued.

The use of oxygen in place of air has always presented certain attractions where this gas is available as a by-product in the preparation of nitrogen by the low-temperature separation of air; but, since mixtures of oxygen and ammonia are explosive, special precautions are necessary for safety of operation. I. W. Cederberg (24) introduces the gas mixture into the converter by way of a series of capillary tubes. Oxidation is effected in the usual way, by means of a transverse, electrically heated, platinum net, following which the reaction mixture is immediately cooled with water or nitric acid. A suitable reaction mixture contains 25 per cent. by volume of ammonia and yields directly nitric acid of about 68 per cent. concentration. The cooling of ammonia oxidation catalysts is also treated by Jost (25), who employs hollow contact bodies, the external surface of which is used to catalyse the reaction while the internal surface is cooled. Mixed oxidation is touched upon in a specification (26) of the I.G., according to which gases containing both ammonia and sulphur dioxide or hydrogen sulphide, together with air, are passed over oxidation catalysts at 500–850° C., with production of a mixture of nitric and sulphuric acids. The raw material employed is such as is sometimes obtained in the washing of industrial gases; and the products are recovered by further oxidation and condensation or scrubbing in the usual way.

#### Absorption of Nitrous Gases

A further patent deals with the absorption of dilute nitrous gases (27). This is stated to be facilitated by mixture with nitric acid, preferably as a vapour, the lower oxides being thus oxidised to higher oxides of nitrogen. Several specifications treat the concentration of nitric acid by processes of the enrichment type. Thus, the solubility of nitric acid in nitrogen peroxide is greatly increased (28) by working at 30 to 60° C. and at pressures corresponding with the vapour pressure of nitrogen peroxide at these temperatures. It should be noted that the reaction is greatly accelerated by the presence of small quantities of certain easily nitrated organic bodies, such as cellulose. In a process for the production of concentrated nitric acid by enrichment, the I.G. dissolve (29), as usual, the dilute acid in nitrogen peroxide, then remove the appropriate layer and cool this to 5° C., when separation into layers once more occurs, the lower of which yields almost pure nitric acid on evaporating the peroxide.

Turning to work on the salts of nitric acid, alkali nitrites may be oxidised directly to nitrates (30) by means of air at an increased temperature and pressure, for instance, at 15 atm., and at temperatures above 150° C., preferably in the presence of a catalyst such as free soda. The conversion of alkali sulphate or chloride into nitrate is receiving increasing attention. Thus, the I.G. describe a process (31) in which these salts are treated with nitric acid at 35°, then cooled to –5° C. and centrifuged, when practically pure alkali nitrate is obtained. The mother liquor may be freed from residual nitric acid by treatment with sulphur dioxide in quantity sufficient to reduce this to nitric oxide (32) which is scrubbed and re-oxidised to nitric acid. G. Fischer and Concordia-Bergbau (33) convert sodium chloride to nitrate by treatment with nitric acid and steam, the latter being preferably present in a quantity ten to fifteen times that of the acid, otherwise nitrosyl chloride and other side products are formed. Separation is effected by fractional solution. The conversion of sodium chloride to nitrate by means of liquid nitrogen peroxide containing a small quantity of water has also been described (34), the salt being moved in countercurrent to the peroxide, when a dry product is finally obtained. The passage of sodium chloride into sodium nitrate, mixed with calcium phosphate, is mentioned

(15) U.S. Pat. 1,704,214.

(16) Trans. Faraday Soc. 1929, **25**, 462.

(17) Brit. Pat. 303,455; THE CHEMICAL AGE, Vol. XX, p. 129.

(18) U.S. Pat. 1,718,420.

(19) Brit. Pat. 310,635; THE CHEMICAL AGE, Vol. XX, p. 545.

(20) Brit. Pat. 304,872; THE CHEMICAL AGE, Vol. XX, p. 213.

(21) *Chimie et Industrie*, 1929, p. 701.

(22) *J. Ind. and Eng. Chem.*, 1929, **21**, 222.

(23) Brit. Pat. 306,382; THE CHEMICAL AGE, Vol. XX, p. 412.

(24) Brit. Pat. 319,548; THE CHEMICAL AGE, Vol. XXI, p. 407.

(25) Brit. Pat. 300,562.

(26) Brit. Pat. 301,232; THE CHEMICAL AGE, Vol. XX, p. 12.

(27) Brit. Pat. 316,735; THE CHEMICAL AGE, Vol. XXI, p. 223.

(28) Brit. Pat. 320,125; THE CHEMICAL AGE, Vol. XXI, p. 510.

(29) Brit. Pat. 320,125.

(30) Brit. Pat. 306,998; THE CHEMICAL AGE, Vol. XX, p. 339.

(31) Brit. Pat. 303,355.

(32) Brit. Pat. 303,351.

(33) Brit. Pat. 308,028; THE CHEMICAL AGE, Vol. XX, p. 383.

(34) Brit. Pat. 310,230; THE CHEMICAL AGE, Vol. XX, p. 507.

by Jost (35). By treatment with phosphoric acid, sodium phosphate is formed, which is caused to interact with calcium nitrate, when a mixture of calcium phosphate and sodium nitrate is produced. The product is intended as a fertiliser; and an excess of calcium nitrate may be used.

Interesting notes on the technical conversion of sodium nitrate into the potassium salt by interaction with potassium chloride are given by F. Chemnitz (36). The potassium chloride should conform to certain stated conditions of purity; and a product is obtained which contains about 0.4 per cent. of sodium chloride and 0.1 per cent. of sodium nitrate. The operation is controlled by the specific gravity of the solution at given stages. Finally, mention may be made of a method, patented by the Anglo-Chilean Consolidated Nitrate Corporation and G. H. Gleason (37) for obtaining nitre in the form of relatively small non-hygroscopic spherical particles, the molten salt being for this purpose sprayed into a cooling atmosphere.

#### Hydrocyanic Acid and Cyanides

The catalytic formation of hydrocyanic acid continues to receive attention. Thus, the I.G. pass carbon monoxide, together with ammonia, over a contact mass containing carbides of iron, nickel or cobalt. The hydrocyanic acid is removed by condensation as ammonium cyanide or by treatment with soda; and the residual gases are reprocessed over the catalyst, which is preferably maintained at a temperature below 600° C., otherwise ammonia is decomposed. A further process, which is stated to give a good yield of hydrocyanic acid, is due to T. Ewan and Imperial Chemical Industries, Ltd. (38). Formamide is passed over a catalyst consisting of alumina, zirconia or thorina at 500° C. It is found preferable to employ a contact mass which has first been sintered. The California Cyanide Company and Dolley (39) propose the preparation of hydrocyanic acid by treating an alkali or alkaline earth cyanide, in the form of a gaseous suspension, with carbon dioxide; further, the I.G. have described a

method whereby this product is obtained by acting on the cyanide of an alkali metal with an aqueous suspension of an excess of sodium bicarbonate. The temperature is raised to about 50° C., and the hydrocyanic acid is removed by evacuating. Under these conditions no polymerisation takes place.

The I.G. has described (40) the preparation of alkali cyanides by acting on the hydroxides, carbonates, sulphates, sulphides and certain other salts of the alkalis, such as the formate or the acetate, with ammonia and carbon monoxide, or gases containing these, at 400–800° C. If the gases have only a low carbon monoxide and ammonia content, iron may be added as a catalyst, as in the Bucher process. Reaction takes place until a certain water content has been reached; and the gases after removal of this water are once more passed over the mass. Other carbon compounds may be used in place of carbon monoxide (41). Cyanates may be quantitatively converted into cyanides by treatment with carbon monoxide at temperatures above 700° C (42); and a process for the direct preparation of cyanamides of the alkaline earths, including magnesium, has been suggested by N. Caro and A. R. Frank (43), according to which the corresponding oxide is heated to 650–850° C. in the presence of gas mixtures which give hydrocyanic acid, for instance, a mixture of ammonia and carbon monoxide, the reaction being carried out either at atmospheric pressure or at an increased pressure. It is interesting to note that catalyst poisons or water may advantageously be added in order to prevent decomposition. Finally, attention may be drawn to a patent of H. B. Kipper (44), in which manganese ore is recommended as an additional catalyst in a process of the Bucher type.

It is not possible, in a short review of the above nature, to deal with every paper and patent specification in this field which has been published during the year; but the above summary may be taken as a synopsis of much of what has been done.

## Chemical Researches Undertaken by the D.S.I.R. Valuable Work for British Industries

*No more important aspect of British chemical industry exists than the research side. In this carefully compiled article a record is given of the chief branches of industrial research that fall within the scope of that admirably directed body the Department of Scientific and Industrial Research, and of the recent progress registered.*

THIS article contains a brief review of some of the chemical investigations in progress in the various research stations of the Department of Scientific and Industrial Research. In addition to the researches carried out in the research stations, the Department also assists specific researches of a fundamental character which are likely to lead to results of general interest and importance. Examples of these in the field of chemistry are: (1) the work of Professor W. A. Bone, of the Imperial College of Science, on gaseous reactions at high pressures; (2) the investigation of the sterols of vegetable and fish oils and fats, by Professor I. M. Heilbron, at the University of Liverpool; and (3) the researches of Dr. I. S. MacLean, of the Lister Institute, into the nature of the processes by which fatty acids are formed from carbohydrates.

#### Corrosion of Metals

A general study of the factors which control the rate of corrosion of a metal in salt solutions has been continued at the Chemical Research Laboratory, and measurements of the initial rate and of the total corrosion of zinc in two similar series of solutions of potassium chloride and sulphate have been completed. By control of experimental conditions, it has been found possible to reach a reproducibility of 1 per cent. from the mean of two duplicate experiments throughout a wide range of solutions. Work has been begun on steel and on magnesium and its alloys along similar lines.

Work on atmospheric corrosion has included the effect of heating zinc specimens in air on subsequent oxidation, the collection and analysis of corrosion products from various

copper structures and an investigation of a method for the determination of small quantities of carbonate in presence of excess of chloride and sulphide. The typical green patina on copper after prolonged exposure to the open air has been found to consist essentially of basic copper sulphate, except in purely marine atmospheres, where basic copper chloride predominates. Time-corrosion curves have been obtained for copper in various synthetic atmospheres.

Considerable progress has been made in connection with researches on the protection of aluminium against corrosion by the electrodeposition of zinc which are being carried out at the Royal Aircraft Establishment, Farnborough, on behalf of the Electro-deposition Research Committee of the Department. Successful experiments have been made on a small technical scale with zinc sulphate and zinc cyanide baths; and test pieces of aluminium, protected by zinc in this manner, have been subjected to severe corrosion tests, with satisfactory results.

Corrosion of hermetically-sealed metal food containers is one of the more important problems of the canning industry. Apart from any question of metallic poisoning, it causes discoloration and deterioration in flavour of the contents of the can, and may result in perforation. The Low Temperature Research Station, Cambridge, has therefore undertaken an investigation of the acid corrosion of iron under conditions approximating to those inside a can of fruit. Preliminary results have shown that different samples of commercial sugars vary widely in their corrosive effects. Sulphur, in the form of

(35) Brit. Pat. 306,046; THE CHEMICAL AGE, Vol. XX, p. 411.

(36) *Chemiker Zeitung*, 1929, 53, 85.

(37) Brit. Pat. 300,369.

(38) Brit. Pat. 305,816; THE CHEMICAL AGE, Vol. XX, p. 258.

(39) Brit. Pat. 299,019.

(40) Brit. Pat. 301,565; THE CHEMICAL AGE, Vol. XX, p. 30.

(41) Brit. Pat. 316,647; THE CHEMICAL AGE, Vol. XXI, p. 223.

(42) Brit. Pat. 307,778; THE CHEMICAL AGE, Vol. XX, p. 383.

(43) Brit. Pat. 286,611.

(44) U.S. Pat. 1,699,362.

sulphur dioxide or as sulphide, increases corrosion, whether air is present or absent; in the form of sulphuric acid, it has little or no effect.

#### Synthetic Resins

Research on synthetic resins has included further studies of the condensation products of formaldehyde with phenolic compounds and a preliminary investigation of resins other than those of the phenol-formaldehyde series, including those prepared from ketones and formaldehyde. The heat-hardening resin made with *m*-cresol has shown very satisfactory properties in regard to freedom from blistering and rapidity of stoving, and at the same time has given breakdown voltage figures of higher values and greater consistency than those of trade resins examined. Experiments on fusible resins from *o*-cresol, *p*-cresol, *m*-cresol and symmetrical *m*-xylenol have yielded crystalline intermediate products which should throw further light on the mechanism of these condensations.

#### Low-Temperature Tars

Tar obtained by distillation of coal at low temperatures differs very considerably in composition from high-temperature tar, and has received comparatively little attention from the point of view of its possible uses in chemical industry. In 1925 it was therefore decided that the Chemical Research Laboratory should undertake, in collaboration with the Fuel Research Station, an exhaustive research on the constituents of low-temperature tars obtained from standard coals carbonised under accurately controlled conditions. These investigations include (1) the isolation and identification of crystallisable chemical constituents; (2) a study of the physical and chemical properties of various resinous derivatives; and (3) technical applications of the tar constituents. The results so far obtained indicate that, broadly, tars produced by low-temperature carbonisation of bituminous coals do not differ widely in their characteristics, and although the proportions of the constituents vary from one tar to another, the same classes of constituents appear to be present in each tar. The crystallisable phenols which, when distilled with water under reduced pressure and subsequently fractionated *in vacuo*, show little or no tendency to redden either alone or in emulsions, can be separated in a form suitable for use in the manufacture of synthetic resins, germicides, disinfectants and wood preservatives. The resinous products, conveniently divided into four groups: (1) neutral resins; (2) phenolic resins or resinsols; (3) acidic resins or resinoic acids; and (4) basic resins or resinamines, can be isolated from tar by simple processes, and are likely to find industrial applications. When dissolved in appropriate solvents and applied to wood surfaces, they furnish lacquers, stains and varnishes. Isolation of the original constituents of low-temperature tars, and particularly of the four groups of resinous products, has been rendered practicable by the use of a solvent method employed at comparatively low temperatures. Owing to this avoidance of high temperature, other decomposable constituents of the tar, such as waxes and higher aromatics, have been obtained in appreciably larger yields.

#### Hydrogenation of Coal

Experiments on the hydrogenation of coal by the Bergius method are being carried out at the Fuel Research Station in an intermediate scale continuous plant capable of treating one ton of coal per day and also in small bombs of a capacity of about 250 grams of coal. The work consists of a close study of the chemical reactions involved, in view of their fundamental importance for elucidating the constitution of coal and the factors which determine its caking properties. It has been found that carbonaceous materials ranging from cellulose and wood to anthracite, and including all types of coal and lignite, can, by controlled treatment with hydrogen under pressure, be converted into material which yields a strong coherent coke on carbonisation.

#### High-Pressure Gas Reactions

During the last twenty years there has been a remarkable development in synthetic industrial chemistry. Gaseous syntheses are being effected to-day under conditions of high temperatures and pressures which twenty-five years ago would have been regarded as quite impossible of industrial application. In view of great possibilities of developments

in this direction, a general investigation of high-pressure syntheses was initiated in the Chemical Research Laboratory in January, 1926. A study has been commenced of interactions occurring between carbon monoxide and hydrogen under conditions of high pressure and temperature, since these reagents offer a wide selection of possible chemical changes and thus provide a favourable introduction to this branch of synthetic chemistry. During the past year further additions, including a super-pressure Hofer compressor, have been made to the high-pressure plant. With this improved equipment the effects of various catalysts on the synthesis of ethyl and other alcohols are being examined. In the carbon monoxide and hydrogen experiments initial experiments with a large scale converter system revealed a tendency in cobalt catalysts to become overheated. While it was found possible to control this tendency in some measure a considerable proportion of carbon monoxide was in all cases converted to methane. Substitution of manganese for chromium in a zinc-cobalt catalyst appears to have a marked effect on the yields of ethyl and higher alcohols and it is proposed to examine simple mixtures containing manganese in the new one-inch system.

#### Water Softening

There are in particular three directions in which rapid progress is being made in this country in the treatment of water. The Metropolitan Water Board is conducting researches on two of these, namely rapid filtration and chlorination. In regard to the third—the base exchange or zeolite process—the mode of action is very imperfectly understood from the physico-chemical point of view, and it has therefore been arranged for an investigation of the process to be carried out in the Chemical Research Laboratory on behalf of the Water Pollution Research Board. A summary of existing knowledge of the process has been published and investigations are now proceeding with special reference to the following points:—The extent of the base-exchange and its variation with the rate of flow of water; the wastage of material; the possibility of the contamination of softened water by silica and alumina; the degree to which the action is a surface one; and the process of regeneration.

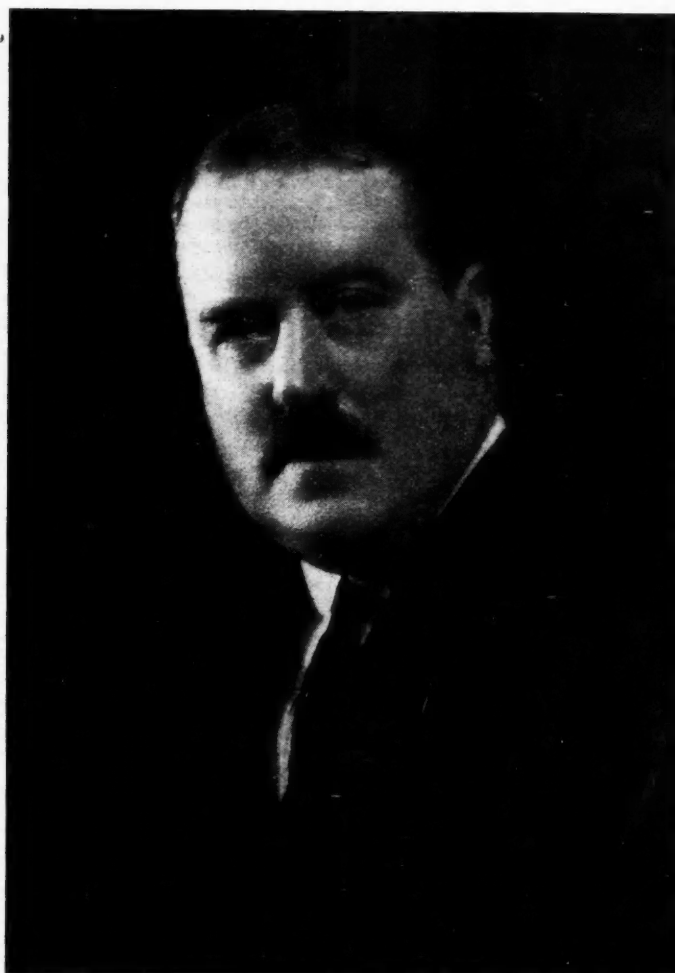
#### Chemotherapy

Investigations are proceeding at the Chemical Research Laboratory in collaboration with the Chemotherapy Committee of the Medical Research Council on the preparation of substances likely to be of therapeutic value, and so far attention has been mainly directed towards the compounds of the Bayer 205 type, containing carbazole, fluorene or fluorene groups as terminal nuclei. The work is necessarily of a purely exploratory character since our present knowledge of the relationship between constitution and trypanocidal activity is too limited and imperfect to enable predictions to be made as to the probable activity of any compound of these series.

Work is also in progress on the preparation of drugs containing antimony which have not hitherto been explored to the same extent as arsenicals. Nevertheless, antimony in certain combinations appears to possess a definite trypanocidal activity, and certain of the compounds already prepared have exhibited a definite though slight action.

#### Free Lime Determinations in Portland Cement

The work of the Building Research Station on the nature of pozzolanas and on the effect of adding them to Portland cement and lime mixes has necessitated the determination of the change in free lime content of pozzolana lime and pozzolana Portland cement mortars. One of the principal uses of pozzolana is to render set Portland cement more chemically inert. Since the pozzolana combines with the lime that is liberated when the cement is hydrated, it is clear that a direct test of the true activity of a pozzolana from this aspect is to determine its effect on the free lime content of a Portland cement mortar. But hitherto the estimation of calcium hydroxide in the presence of either unhydrated or hydrated Portland cement has not been satisfactorily achieved. The work is not yet finished, but it is apparent that two methods of estimating free lime will be needed; one to be used when hydratable matter is present as with all mixes of cement, and the other for mixtures of calcium hydroxide and pozzolana when the amount of lime may be as high as 30 or 40 per cent. The first may be provided by an adaptation of the thermal method, whilst for the second an electrometric device may be used.



PROFESSOR JOCELYN F. THORPE, F.R.S., D.Sc., etc.

*President of the British Chemical Society, Professor of Organic Chemistry at the Imperial College of Science and Technology; an honoured and well-known figure in international chemistry.*

In the course of this work it has been found that pozzolanic activity is affected to a large extent by change of temperature. Thus while crystalline silica is practically incapable of combination with lime at 15° C., at temperatures not very much higher its activity is quite definite. This suggests the possibility that a pozzolana of appreciable value in warmer climates may be of little value in countries with low mean temperatures.

#### Freezing of Colloids

Recent work carried out on the freezing of muscle and egg yolk at the Low Temperature Research Station has emphasised the importance of the concentration of electrolytes in determining the degree of reversibility and the presence of critical temperatures in the freezing of complex colloidal systems. With these facts in mind, a study of the effects of freezing on simple systems of pure colloids, both in the presence and absence of added salts, has been initiated. After many trials, it was found that a suitable system for a study in freezing consists of a mixture of carbon tetrachloride and heptane (density=1) dispersed in water with kaolin as the emulsifier. Preliminary experiments with this emulsion have shown that

in all probability critical freezing-temperatures and critical salt-concentrations do exist, but so far it has not been possible to eliminate completely the "rate of freezing factor."

#### Chemical Work on Fruit

Two sets of observations on the change in the chemical composition of apples during storage have now been completed at the Low Temperature Research Station. The apples have been grown in plantations which have been studied as regards the chemical and physical properties of their soils, and have known manurial histories. Chemical analyses of representative samples from each plantation have been made at the Imperial College of Science, both at the time of gathering and after 100 days' storage at 34° F. While interesting from the point of view of the study of the course of senescence, the chemical data obtained do not indicate any clear relation between chemical composition (percentage content of sugars and acid) at the time of gathering and the keeping quality of the fruit in storage. On the other hand, the data do indicate that the bad keeping apples lose acid and sugar on respiration more rapidly than the better keepers.

## The British Synthetic Fertiliser Industry Steady World Expansion

*The growth of the synthetic fertiliser industry in this country represents one of the most important developments of recent years. Its progress and possibilities are clearly discussed below.*

THE past year in the synthetic fertiliser trade has been marked by steady and substantial progress. The value of synthetic fertilisers is being more widely appreciated each year, and each year shows a corresponding increase in the manufacture and use of these products.

Synthetic ammonium sulphate was first manufactured in this country on the commercial scale in 1923, and during the past year Imperial Chemical Industries has completed more large units at Billingham, thus bringing the production of ammonium sulphate to nearly three times the production in 1928. The bulk of the increased output has been shipped to the Near and Far East, but sulphate in increasing quantities has also been exported to all parts of the British Empire.

The phenomenal growth of Billingham from a small village to what is really a collection of factories under one management could not have been achieved without a great amount of research. With the completion of the original construction programme, research is now being directed towards the improvement of manufacturing processes and towards future developments. Attention is being given to such matters as the behaviour of fertilisers under abnormal physical conditions, which is being studied in a large chamber where temperature, pressure and humidity can be varied indefinitely. In this way the products can be tested under all the climatic conditions to which they are likely to be subjected during their shipment to any part of the world; the information thus obtained enables the consumer to receive his goods in the same excellent condition as they leave the factory. Agricultural research work on the application of fertilisers is being carried out simultaneously, but this will be dealt with later. Meanwhile it is interesting to note that in spite of the dry season, the advantages of the use of ammonium sulphate have never been more clearly demonstrated.

#### Nitro Chalk

Nitro chalk, the new British fertiliser that contains half its nitrogen as nitrate and half as ammonia, has met with such success that a new plant which will increase the present production is already in course of construction. Nitro chalk possesses many advantages as a top dressing. It contains 15.5 per cent. nitrogen and 48 per cent. carbonate of lime. The nitrogen existing as nitrate is absorbed by crops more rapidly than the nitrogen existing as ammonia, so that the fertiliser is not only rapid in its action but lasting in its effect, while the carbonate of lime helps to keep the soil sweet.

Nitro chalk was first produced as a powder, but has been marketed this year in granular form, an improvement which has made it extremely popular with farmers. The hot mixture is sprayed down a high tower and the granules thus formed

are carefully graded to give a uniform product. Granulation enables the manufacturer to keep the quality of his product constant and to deliver it in a free running condition, easy to sow by hand or machine. The granules dissolve more slowly than the powder, with the result that the fertiliser is not washed away by heavy rain, and possesses the further advantage that it does not stick to foliage. Nitro chalk is sold both in this country and abroad. It is already being used in such countries as Spain, Egypt and Palestine, while promising results are anticipated from tests at present being carried out in other parts of the world, e.g., New Zealand.

#### New British Compound Fertiliser

The latest concentrated artificial fertiliser to be manufactured in England is ammonium phosphate. A plant to make this fertiliser has been completed at the Billingham works, and production on a large scale has just begun. The plant can produce a fertiliser containing either 45 per cent.  $P_2O_5$  with 14 per cent.  $N_2$  or 18 per cent.  $P_2O_5$  with 18 per cent.  $N_2$ . With these two products the farmer will be able to obtain a material to suit his own special requirements, while only the addition of a potash salt is required to provide a complete fertiliser furnishing N, P and K in any desired ratios. Since normal superphosphate contains from 16 per cent. to 18 per cent.  $P_2O_5$  and no nitrogen, while ordinary farmyard manure contains from 0.2 per cent. to 0.4 per cent.  $P_2O_5$  and from 0.45 per cent. to 0.65 per cent.  $N_2$ , the saving in freight and cost of spreading by the use of the new fertiliser is obvious. When the product has to be shipped to distant parts of the world and large quantities have to be applied to the land the actual monetary value of this advantage is very considerable.

The fertiliser is being produced as white crystals, in which the phosphate is completely soluble both in water and in citric acid. It is entirely neutral and contains no free phosphoric acid to rot the bags or render the soil acid. In addition, ammonium phosphate does not revert easily in the soil, so that crops are able to derive the maximum benefit from its use.

#### Agricultural Research

The research organisation created for the synthetic fertiliser industry by Imperial Chemical Industries was completed this year, when their agricultural research station at Jealott's Hill was formally opened by the Rt. Hon. J. H. Thomas, P.C., M.P., on June 28. The station consists of the most up-to-date laboratories and two farms where the results of laboratory research are tested on both arable land and pasture, and the effect of scientific treatment on crops and stock is carefully recorded. Investigations are being carried out both of a fundamental nature, applicable to all parts of the world,

and of a specialised nature applicable to any particular problem that may arise in this country or abroad. Jealott's Hill has a two-fold function; firstly, to aid in the scientific development of agriculture in Great Britain; and, secondly, to assist the agricultural development of the Empire.

The staff of the research station, though for the most part located here, is also equipped as an overseas expeditionary force, to go anywhere within the Empire or without, while advisers are already stationed in different parts of this country and the Empire to whom any farmer may apply for free advice. The Agricultural Research and Advisory Department of Imperial Chemical Industries aims at close co-operation between its staff and the staffs of other research stations; it endeavours to attain this aim by assisting in schemes involving co-operation in research by all who have the interests of agriculture at heart, and by making grants to the general purposes of the chief agricultural research institutions of this country. Field and other trials are carried out by Area Trial Officers who work in co-operation with farmers, while the Agricultural Intelligence Department of Imperial Chemical Industries is responsible for the dissemination of agricultural information, and collects suggestions with respect to new problems at home and overseas which would appear likely to repay investigation.

During the year, Sir Frederick Keeble and other members of Imperial Chemical Industries Agricultural Research and Advisory Department have visited various parts of the world to investigate the possibility of the improvement of all kinds of farming by means of artificial fertilisers. Their investigations have confirmed the expectation that there is almost unlimited scope for increase in the scientific use of fertilisers. Special interest attaches to the work on intensive cultivation of arable land and grass land in Canada, Australia, Africa and the Near and Far East, and in India, where a very large potential market exists for ammonium phosphate, which has been shown to be of great benefit to the rice fields.

Intensive cultivation is being successfully applied to cotton and tobacco, to sugar and tea, to wheat and barley, while such diverse crops as banana, citrus, vine, coconut, coffee and pineapple are receiving equally close attention. All this work is opening up new markets and creating new demands for fertilisers. The farmer who does not avail himself of the new knowledge will find himself unable to compete with the farmer who does, and consequently the more fertilisers that are used, the greater will be the demand for yet more fertilisers. Imperial Chemical Industries' policy of looking ahead will enable Great Britain to meet that demand as fast as it materialises.

## The British Dyestuff Industry in 1929

### I.—Technical Progress: By L. J. Hooley

*The scientific and technical progress of the British Dyestuffs Industry is discussed by Mr. L. J. Hooley (of Scottish Dyes, Ltd.) a well-known and valued contributor to our columns, and the notes on the situation from the colour user's standpoint complete the review.*

As the end of the Dyestuffs Act approaches more closely, the question of the efficiency and position of the British industry becomes of greater and greater interest. There is now not much more than another year of the original ten to run, and what is to happen then does not seem much nearer determination than it did this time last year. There does not appear as yet to be any general desire to leave the industry to fight its battle entirely alone. The attitude of the Government is likely to be a deciding factor, and although on the side of principle its pronouncements have been against rather than for protective and safeguarding measures, it has not yet set any precedent in practice. The chemical industry has, however, shown such determined and enlightened policy in its recent activities, and the dyestuffs section has made such excellent use of the protection which has been afforded, that it can take credit for having helped itself to the utmost.

#### Substantial Increases in Production

Coming to the question of recent progress, what facts are there to consider as showing continued achievement? One of the most gratifying is the increased production. The figures for the last three years, as given in the Board of Trade returns, are: 1926, 30,297,000 lb.; 1927, 39,551,756 lb.; 1928, 50,907,080 lb. These represent an increase of 29 per cent. in 1928, and 30 per cent. in 1927. The production figures for 1929 are not likely to be available for some time yet, but there is no reason to assume that any decrease will be shown.

In order to arrive at a fair estimate of the significance of the above production figures the import statistics should be considered as well. For the same three years these are: 1926, 4,232,587 lb.; 1927, 4,990,356 lb.; 1928, 5,030,571 lb. It will be seen that although on the increase, the rate is less than that of the increase in production, so that the proportion of the trade going abroad is decreasing, the percentages of home production for 1928, 1927 and 1926 are, in fact, respectively 91, 89, and 87 per cent. The percentages imported are therefore 9, 11 and 13 per cent., showing a proportional reduction of nearly one-third. It must be remembered that it is only the considerable enterprise shown by the foreign producers that has kept the import figures as high as they have been.

#### Prices

Next as regards prices, the factor has again been reduced, this time from 2 down to 1.75. This 1.75 is a maximum and not a minimum figure. The most interesting fact in connection with prices, however, is that given by Sir Henry Sutcliffe

Smith in his survey at the last annual meeting of the Colour Users' Association.

Sir Henry Smith, while maintaining that, compared with the general level of commodities, dyestuff prices were too high, stated that inquiry had shown that the prices ruling for dyestuffs in this country were not substantially higher than those paid by users on the Continent, and while on the average these were lower in Holland and Germany, they were actually higher in other countries.

#### Exports

Turning to exports, this is a factor which affords the severest test of the industry, for here British manufacturers are in open competition with the world. While admittedly the position of the United Kingdom is still not satisfactory, the following figures, abstracted from the latest report of the United States Department of Commerce, show that considerable advances are being made, for the U.K. figures show the largest relative increase, and the ratio of exports to production is now approximately the same as in the U.S.A.

#### EXPORTS IN DOLLARS VALUE.

Year.	Germany.	Switzerland.	France.	United Kingdom.	United States.
1928 ..	54,700,000	15,100,000	2,400,000	3,900,000	6,500,000
1927 ..	55,500,000	14,600,000	3,100,000	3,000,000	5,500,000
1926 ..	47,100,000	12,000,000	5,900,000	2,400,000	5,900,000

It is to be expected that the formation of Imperial Chemical Industries should prove a factor in raising the export figures. The policy of the company is in one respect sufficiently indicated by its title; and while even with regard to dyestuffs alone it may be expected to be in a better position than could have been any of its constituent dyestuff companies, the fact that dyestuffs only form a part of its chemical activities makes the establishment of foreign departments all the easier.

Indications show that the volume of exports is still increasing, and that 1929 should show a further considerable advance.

#### Conditions Abroad

There is not room for more than a passing reference to the progress of other producing countries. The United States made a new record with 95,000,000 lb. in 1928, with vats (excluding indigo) rising from 5,961,000 to 6,300,000 lb.; in the early part of the year comment was aroused by the fact that imports were increasing. France remains steady, Switzerland shows increased progress at home and abroad. Germany appears to be stationary at home and improving abroad. Italy is extending production to the vats and other of the

more select series. Japan is receiving further subsidies, and also progressing satisfactorily with extensions.

Of general interest is the signing of the sales agreement by the five major producing concerns of Germany, France and Switzerland, which gives permanence to the informal agreement of the last three or four years.

#### Research

On the technical side, there has been the continued intense research carried out during the year by the principal manufacturers. The I.G.F.A.-G. again lead the list, but the contribution of the British firms is again larger in 1929 than in any previous years. Full details will be found in the various issues of the DYESTUFFS MONTHLY SUPPLEMENT of this journal.

#### New Colours

Technical developments become of greatest interest to the user when they reach the stage of new colours, and 1929 has a good number of these to show.

J. W. Leitch and Co. have brought out Naphthol J.W.L. among other products, for their methods of dyeing silk and wool, without caustic alkali. Among new announcements may

be mentioned L.B. Holliday's Paradone Grey R, a new all-round fast colour of their vat series, Monochrome Olive Brown G, and Fast Chrome Worsted Blues B and G.

From the British Dyestuffs Corporation and Scottish Dyes, Ltd., several additions have also come. Acronol Green is a new basic colour suitable for fast shades on acetyl silk. Five new reds, making eight in all, and two browns, namely, the R and G brands, have been added to the Duranol range. Red G is a further member of the new Icyl series for the level dyeing of silks of viscose type. Solway Blue BGA is an excellent levelling colour, particularly suitable for mode shades, and Blue RS is a similar type, which can be used with chrome colours. Finally, there has been Fast Scarlet GS for cotton, Lake Scarlet GS for printing inks and paper-surfacing colours, Leather Green for motoring cloths, Naphthalene Fast Navy H (a new acid colour with excellent washing fastness), and Caledon Red GG, a hot dyeing red with good tinctorial properties.

In summary, it may be said that the British industry is in a very vigorous condition, and the figures for production, imports, exports, and technical advances are better than at any previous time in its history.

## II.—The Colour User's Point of View

A REVIEW of the work of the Colour Users' Association during the past year once again throws light on its many and varied activities on behalf of the colour using industries.

Perhaps the most important feature has been the reduction of the factor from 2 to  $1\frac{1}{2}$  times the pre-war price in dealing with applications for licences on price grounds. This result has been achieved by negotiations between the representatives of the dye makers and the colour users on the Dyestuffs Advisory Licensing Committee, and has naturally given considerable satisfaction to consumers throughout the country. On this occasion, as on every other when a reduction of the factor has been made, the alteration was unanimously agreed to by both parties on the Licensing Committee.

#### Licensing Procedure

Several points of interest have arisen in connection with the operation of licences on price grounds. For instance, it is still contended by colour users that where a British manufacturer introduces an equivalent for certain foreign products, the British user should not be called upon to pay a minimum of  $1\frac{1}{2}$  times pre-war, if, prior to the introduction of the British product, the foreign price was less. It seems an anomalous position that users should be faced with the prospect of an increase in prices simply because British equivalents have been introduced, even although such instances are not numerous. Representations have, therefore, been made to the Licensing Committee that in such cases the British manufacturer should accept, for a minimum period of six months, the same price as foreign manufacturers have been selling at during the previous three months.

Another point in connection with licensing procedure that has engaged the Council's attention is the suggestion that in dealing with applications on price grounds the British manufacturer should be entitled to make an addition to the pre-war price if, in his opinion, the quality of the product offered was superior to that manufactured pre-war. The view put forward by the users' representatives is that this practice involves a breach of the principles governing the factor method of dealing with price applications, which has always been operated on the understanding that the factor represents a maximum figure.

In last year's review, reference was made to the handicap users were under owing to the absence of competitive quotations. The question of the modification of the procedure in connection with price applications is again being taken up and the Licensing Committee urged to accept the suggestion that in such cases the foreign suppliers' quotations shall only be submitted to the chairman and neutral members of the Licensing Committee.

During the year a change was made by the Government authorities in the regulations governing the importation of dyestuffs samples to the effect that such samples could only be imported by parcel post. This alteration entailed con-

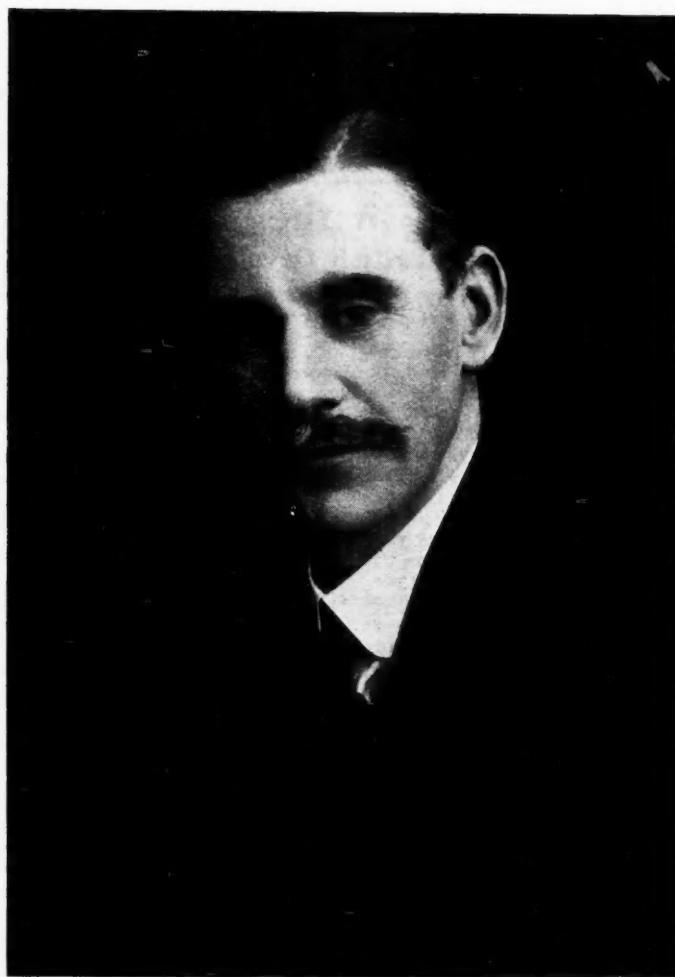
siderable expense, delay, and inconvenience for the consumer, and after strong representations had been made to the Board of Trade an order was issued by which the importation of dyestuffs by sample post was again authorised, and it is hoped that these regulations will continue in force without further restrictions.

#### A Continental Inquiry

The Association has again devoted a great deal of time and attention to the question of dyestuff prices and the world dyestuffs situation. An informal deputation has recently made private inquiries into the present dyestuffs situation on the Continent, from which it has been established that conditions abroad are now more stable and that the close competition of a few years ago has given way to a series of price conventions which have had the effect of regulating and maintaining prices, and that the present tendency abroad is towards organised control and a definite price policy. The inquiry also established that British dyestuffs prices generally compare favourably with those ruling on the Continent. Whilst this is encouraging, it is distinctly disturbing to reflect that world dyestuffs prices are still too high as compared with the general price level of wholesale commodities. Two factors are probably the cause of this anomaly: (1) the productive capacity is considerably in excess of the consumption, and (2) the effect of international price conventions.

#### Safeguarding Duties on Chemicals

Once again the Association has made application for the exemption of oxalic acid from duty under the Safeguarding of Industries Act, and exemption has been granted for a period expiring in December, 1930. It seems anomalous that a product not made in this country should be scheduled for protection under an Act placed on the Statute Book ostensibly for the purpose of imposing "duties of customs on certain goods with a view to the safeguarding of certain industries and the safeguarding of employment in industries in the United Kingdom." The onus is thus placed upon the consumer of obtaining exemption from such duty of a product which is not made in this country and which apparently should not be scheduled under the Act. In such cases as this it would seem more equitable to delete the product from the schedule, and thus relieve the consumer of the formality of applying for exemption, leaving it for any prospective British maker to apply for inclusion if and when production commenced in this country. The position of consumers with regard to formic acid and one or two other chemicals included in the Schedule of Safeguarded Chemicals continues to be unsatisfactory. While users fully recognise the necessity for efficient producing units of important chemicals in this country, they nevertheless feel that an obligation rests on the home producer to sell a protected product at somewhere near the world market price.



MR. J. ARTHUR REAVELL, M.I.MECH.E., M.I.CHEM.E.,

*Managing Director of the Kestner Evaporator and Engineering Co.,  
and President of the Institution of Chemical Engineers, who contributes  
an article on progress in the field of chemical plant manufacture.*

The Association's Chemical Price Bulletin is now issued quarterly and has been considerably enlarged. The information given continues to be highly appreciated by members.

The Association has taken a keen interest in the formation of the proposed British Colour Council, and is directly represented on the management committee. Members of the Association's Council have also been independently elected to the management committee, so there is every prospect that when the proposed Colour Council commences its activities, the members of this Association will be kept posted with all the latest developments.

#### The Forthcoming Dyestuffs Report

In connection with the approaching expiry of the Dyestuffs Act a committee of the Association is engaged on the preparation of a report for submission to the Dyestuffs Industry Development Committee of the Board of Trade. The lack of any official statistics regarding the dyestuffs industry in this country is a great handicap in attempting any com-

prehensive survey of the dyestuffs situation, and repeated representations have been made to the authorities regarding the advisability of the Board of Trade compiling and issuing complete annual reports on production in this country. As pointed out by the chairman (Sir Henry Sutcliffe Smith) in his annual meeting address, it is surely extraordinary that those who desire detailed information on the subject of British dyestuffs must refer to American publications to obtain it.

In accordance with his usual practice, the chairman gave an address at the annual meeting which consisted of a comprehensive survey of the dyestuffs position. These annual addresses have been a feature of Sir Henry Sutcliffe Smith's term of office, and together they form a very valuable commentary on the situation for the past few years. Each year a report of the proceedings has been printed and circulated widely, and further copies may be obtained at any time on application to the secretary.

## The Year's Progress in the Solvents Industry

### Important British Developments

*The following article, written by an eminent authority in close contact with the solvents industry, deals generally with the subject, touching, among other things, on acetone, butyl alcohol, solvent recovery and numerous other topics of interest.*

THE importance of the solvents industry can be measured by the enormous increase in the production of aliphatic chemicals that has taken place in the United States between 1921 and 1929, the quantity having increased from 20 million pounds to nearly 300 million pounds, of which about two-thirds appears to be used in the lacquer industry. The production of finished aromatic chemicals in the same interval increased only about three-fold. There is no doubt that the production of aliphatic chemicals has considerably increased in this country also during the same period, but the increase cannot be compared with that which has taken place in the States, and there is a danger that America may win a pre-eminence in the field of aliphatic chemicals similar to that once held by Germany in the coal-tar dyestuffs industry.

#### Development of Well-Tried Solvents

No important development has taken place during the year in the production of new solvents, and many of the new solvents which have been put on the market and advertised largely during the past few years seem to have made very little progress. The demand for the old and tried solvents, such as the ketones and the esters, remains steady, and in many cases has increased considerably. Among the new ones, only the glycol ethers and their derivatives seem to be used to any extent. These are being imported into this country, but still in relatively small amounts. According to a review of the chemical industries of Yorkshire (*THE CHEMICAL AGE*, 1929, 21, 277), ethylene glycol is being produced in that county from ethylene obtained from ethyl alcohol. The processes used in Germany and America for the manufacture of ethylene glycol were described by Schrader in the *Zeitschrift für Angew. Chem.* (1929, 42, 541); in the same article some figures on costs and production are given, and properties and uses of glycol and some of its derivatives enumerated. The physical and chemical properties of 1:4-dioxan (diethylene oxide) are described, and the potential value of the compound as a lacquer solvent discussed, by Reid and Hoffmann in *Industrial and Engineering Chemistry* (1929, 21, 695). Several patents have been taken out during the year for the production and use of the glycols, glycol ethers, and other glycol derivatives, but few of these present any feature of special interest. A series of patents granted to the I.G. Farbenindustrie for the manufacture of the butylene glycols are perhaps the most noteworthy.

A few papers of interest have appeared in American and German journals on the properties of lacquer solvents. R. Calvert, in *Industrial and Engineering Chemistry* (1929, 21, 213), has made a very useful contribution as to the bearing which the composition of a solvent has on its solvent properties, particularly with regard to the cellulose esters. A comprehensive survey of modern cellulose acetate lacquers was published by Reid and Hoffmann in *Industrial and Engineering Chemistry* (1929, 21, 958), in which the principal properties of each of the solvents in tabulated form is given, and the

different types of solvents and mixtures of these are discussed at length. An important paper on the flash points of solvents and plasticisers was published by E. von Muhlendahl (*Farben-Zeitung*, 1929, 34, 1427). The various methods for determining flash points were critically discussed and the flash points of a large number of solvents, plasticisers, and characteristic binary and ternary mixtures of these were redetermined and tabulated, using the Ormandy-Craven flash-point apparatus. The effect of thinners on the viscosity of nitrocellulose solutions was discussed by B. Pain (*J.S.C.I.*, 1929, 48, 2237) from a physico-chemical point of view.

#### British Production of Acetone

The most important development in the production of acetone has undoubtedly been the manufacture in this country of large amounts of this compound by a synthetic process from ethyl alcohol. The development of this process is due to British Industrial Solvents, who have established a factory on a large scale in Hull for the manufacture of acetone and other compounds. Several patents of the Consortium für Elektro Chem. Industrie claim the production of acetone directly from acetic acid by catalytic treatment, but none of them seem to contain any novel feature, with the possible exception of slight modifications in plant construction. With regard to ketones other than acetone, the Société Lefranc have made a number of claims as to the value of "ketol" as a solvent, but they do not seem to have put on the market any appreciable amount of the product. An interesting patent has been taken out by the I.G. Farbenindustrie for the production of higher ketones by the catalytic conversion of dihydric alcohols. High yields of methyl ethyl ketone are said to be obtained from butylene glycol.

#### Butyl Alcohol

The synthetic production of butyl alcohol in this country has now become an established fact, and butyl alcohol produced from ethyl alcohol will soon be available. A number of new patents have appeared in this field, but none of them present any outstanding feature of interest, being simple modifications in detail of plant construction or in minor aspects of the processes involved. The synthesis of butyl alcohol still depends, and must always, on the classic work of Sabatier.

Although for the moment butyl alcohol is not produced by fermentation methods in this country, a very interesting development has taken place in improving the technique of these processes. This has been patented in the names of Dr. Hutchinson and the Distillers Co., and may become of importance in the British colonies. The essential feature of the new process lies in the fact that it has been found that inorganic nitrogen can serve as a nutrient for the organisms producing butyl alcohol and acetone, and this will allow of the use of a large number of raw materials poor in protein, such as manioc, potatoes, rice, etc. Hitherto these have been considered unsuitable for the butyl alcohol fermentation. Another

important effect of the use of inorganic nitrogen as nutrient is that the yields of both butyl alcohol and acetone are increased, and little or no production of ethyl alcohol takes place. In a modification of this process, which has also been protected in most of the countries of the world, the relation between the amount of solvents produced, namely, butyl alcohol and acetone, can be controlled by the judicious use of ammonium salts of organic acids.

According to an article in *THE CHEMICAL AGE* (1929, 20, 438), the production of butyl alcohol and acetone by fermentation methods has now been developed in Italy. A number of experiments on the use of cane and beet molasses as a raw material for the butyl alcohol fermentation are described by Mezzadrolì and Magno, but insufficient working details are given to judge of the value of the results. An interesting paper on the mechanism of the butyl alcohol fermentation appeared in the *Journal of Biological Chemistry* (Stiles, Petersen and Fred, 1929, 84, 437), in which experiments determining the amount and nature of acids produced during different stages are described. The effect of organic and inorganic acids on this fermentation was also discussed in a paper read at the meeting of the Canadian chemists in Toronto (*Canadian Chem. and Met.*, 1929, 13, 172). Several patents have been published relating to minor modifications of the usual fermentation process, utilising maize, and an interesting one in this connection is United States Patent 1,696,022, which describes a process by which the fermentation is kept free from infecting organisms by the use of antiseptics such as butyl phenol or butyl resorcinol, it being claimed that the butyl-producing organisms become rapidly acclimatised to the derivatives of butyl alcohol although these are extremely toxic to other organisms.

A new organism has been described in America and named *B. Saccharobutylicum*  $\beta$ , which is said to produce butyl alcohol and propyl alcohol, in substantially the same proportions as butyl alcohol and acetone are produced in the normal fermentation. It has also been claimed in other American patent specifications that much higher yields of fusel oils can be obtained in the alcohol fermentation without affecting the yield of ethyl alcohol produced, by the use of certain wild yeasts. An article by Parisi in the *Annali di Chimica Applicata* (1929, 19, 234) rather tends to show that increasing the production of higher alcohols in alcoholic fermentations is not an economic proposition.

#### Higher Alcohols from Cracking Gases

Little work of outstanding importance on the utilisation of cracking gases for the production of higher alcohols has been published. Some interesting results on the action of sulphuric acid on olefines are given by W. Ormandy and E. C. Craven (*J.S.C.I.*, 1929, 48, 203r). Pilat and Minkler (*Przemysl Chem.*, 1929, 13, 185) have investigated the relation between cracking temperatures and yields of olefines, and the yields of higher alcohols that can be obtained from the olefines produced. In the utilisation of cracking gases for higher alcohol production, as well as in the synthesis of higher alcohols from water gas, the purification of the products obtained is one of the most difficult problems, and no satisfactory solution seems to have been found in either case. An interesting review of the production of amyl alcohol from pentane was given in *Industrial and Engineering Chemistry* (E. Ayres, 1929, 21, 899), and several patents describing improvements in the process have been taken out in America.

The manufacture of synthetic methanol from water gas has increased considerably in the United States of America, and manufacture on an industrial scale has already commenced in France, Belgium and Italy. The wood distillation industry, however, in America and on the Continent seems to be holding its own, and the production of methanol by the Tennessee wood distillation plants has recently considerably increased. Large plants for wood distillation have also been started up in Canada.

A number of interesting articles on the changes involved in the synthesis of methanol have been published during the year. Brown and Galloway (*Industrial and Engineering Chemistry*, 1929, 91, 301) have shown that dimethyl ether is a by-product of the reaction, and is formed at temperatures between 350 and 400° C. The action of iron catalysts on mixtures of carbon monoxide and hydrogen has been studied by Audibert and Raineau, but the results obtained were not very conclusive. Further work has also been carried out by

Frolich on the relative value of different catalysts for methanol synthesis, and has been published in various American journals. The number of patents relating to the synthesis of methanol and higher alcohols from water gas continues to increase, but few of the recent ones present any outstanding features of interest. The production of higher alcohols from water gas seems to have made very little progress indeed, and the chief difficulty seems to be the separation and purification of the complicated mixtures of products which are always obtained. The most interesting patent in the field is that of Morgan and Taylor, British Patent 313,061, in which it is claimed that considerable amounts of ethyl alcohol and other products can be obtained from water gas using a special cobalt catalyst.

#### Chlorinated Hydrocarbons

The use of chlorinated hydrocarbons, such as trichlorethylene, dichlorethylene, etc., does not seem to have made any appreciable progress in this country, although it is stated that they are being largely used in America as solvents, insecticides and fungicides. According to an article which appeared in the *Seifen Zeit.*, ethylene dichloride is being used largely in Germany to replace ether in the extraction of fats and oils. The great objection to the use of the chlorine derivatives of ethylene is their toxicity. Many patents, principally of German origin, have been taken out for the production of various chlorinated derivatives of ethylene and its homologues, and in almost all these acetylene is the starting point. The conversion of tetrachlorethane or acetylene tetrachloride into trichlorethylene by passage over absorbent charcoal at 300° C. forms the subject matter of one of the most interesting of these patents (Wacker Ges. für Elektro Chem. Ind., German Patent 464,320).

#### Esters

Many aliphatic esters of a very high grade of purity are now being manufactured in this country, and British Industrial Solvents are among the leading producers of them. Some esters of acids of high molecular weight have been proposed as plasticisers in the lacquer industry, and the ethyl ester of abietic acid has been found to have pronounced solvent properties for nitrocellulose and may perhaps become of importance. It has been claimed that esters of adipic acid and esters of the type methylphenoxy phthalate are valuable plasticisers. A large number of patents relating to the manufacture of esters have appeared, but few of these present any marked features of novelty. United States Patent 1,694,559 (Commercial Solvents Corporation) describes a process for the manufacture of ethyl lactate by heating a mixture of ethyl alcohol, lactic acid and benzol in the presence of anhydrous aluminium salts, and United States Patent 1,708,640 describes the production of esters by passing alcohols in the vapour phase over dehydrogenating catalysts at moderately high temperatures. Other interesting ester patents are those of Imperial Chemical Industries (British Patents 307,137 and 313,316), in which esters of organic acids are obtained by the reaction between organic acid amides and alcohols in the presence of catalysts. The continuous production of esters is described in patents recently granted to the Holzverkohlungs Ind. A.-G.

#### Solvent Recovery

Very little of importance has been published during the year on the question of solvent recovery, but what will probably be the largest solvent recovery plant in Europe is being installed in this country at Stowmarket. It is believed that the process used will be that already employed by the I.G. Farbenindustrie, and that its principal function will be the recovery of acetone. A new process for recovering acetone has been patented by W. Ormandy (British Patent 308,885) in which strong sulphuric acid is used as the absorbent. The I.G. Farbenindustrie have claimed in a patent (British Patent 302,297) that the high-boiling by-products obtained in methanol synthesis have valuable properties in solvent recovery. An interesting article on the health risks from solvent vapours appeared early in the year (*J.S.C.I.*, 1929, 48, 148), and an extremely good account of the difficulties experienced in solvent recovery and the chief faults of modern practice in this direction are discussed from an engineering point of view by Piatti (*Chem. Apparatur*, 1929, 16, 433).

An important conference on vapour absorption and adsorption was held by the Institution of Chemical Engineers at Burlington House early this month, at which several very interesting papers were read.

## Progress Among Chemical Plant Manufacturers

By J. Arthur Reavell, M.I. Mech.E., M.I.Chem.E.

*The following practical and useful notes on improvements and progress in the manufacture of British chemical plant and in the raw materials of the industry are contributed by Mr. J. Arthur Reavell, a very well-known personality in the chemical engineering world, and, incidentally, President this year of the British Institution of Chemical Engineers. Mr. Reavell, it will be noticed, insists on the high standard of merit of British chemical plant productions.*

THE constant advance in science is making new demands and raising new problems for the chemical engineer, and the end of the year seems the proper time to examine what new developments have been made in the industry.

The British Chemical Plant Manufacturers' Association is a body that comprises most of the important makers of chemical plant, and an account of their activities and latest developments during the past twelve months is of very great interest. The old idea that progress is made only on the Continent and in America must be banished, and it is as well to remind ourselves of the enormous amount of research and experimental work that is being carried on in this country to-day. As British chemists have made enormous strides, so have British chemical engineers kept up with and maintained the enterprise of their chemical colleagues.

### International Electrolytic Plant Co.

Much good work has been done by the International Electrolytic Plant Co. in the perfecting of their electrolytic oxygen- and hydrogen-producing plants. One of the difficulties experienced in the past in regard to apparatus of this kind was the problem of maintaining a constant level of electrolyte in the cells—that is, a constant level irrespective of the quantity of gas that is being produced. This company has now designed an apparatus where it is not only possible either to raise or to lower the height of the electrolyte in the cells by operating a single valve, but, having once made this adjustment, the apparatus remains entirely automatic, and is so perfect in its operation that the batteries can be run for long periods without attention, so long as there is no load on them.

It is well known that there is a considerable loss due to entrainment of the electrolyte by the gases, and this also has been overcome by a gas-washing apparatus, whereby all the gas leaving the cells is washed by distilled water on its way to the cells. This method of washing prevents any loss of electrolyte by entrainment with the gases.

The gas-washing apparatus is adjustable and, in addition, there is a device incorporated with the washer to regulate the back pressure on the cells, so as to maintain the same pressure on both sides of the diaphragm.

This arrangement comprises a series of coils so placed in each cell that the temperature is evenly controlled throughout the cell, and either steam, hot water or cold water can be circulated through the elements as required. The important result achieved is that the electrolyte is always at a temperature corresponding to its maximum conductivity, irrespective of the load the cells are carrying. Thus, when the current passing through the cells is small, they are heated with steam or hot water, and the maximum efficiency is maintained, while, when the load is high, the temperature is kept down to a reasonable limit by circulating cold water.

A further advantage is that by passing steam through the elements before the battery is put into circuit, the electrolyte can be quickly raised to the correct temperature. This avoids the waste of power which otherwise occurs when the cells are put into circuit cold, until the electrolyte has reached normal working temperature.

During the past year the company has shipped a large number of plants to all parts of the world.

### Huntington and Heberlein

The headway made by Huntington and Heberlein in the development of their mechanical furnaces for roasting pyrites has resulted in their constructing and supplying the largest furnaces of this kind ever built in Europe. The first three furnaces of this type are fitted with twelve super-imposed roasting shelves, commanded by a central air-cooled revolving shaft, on which are mounted the air-cooled rabbling arms. As the concentrated heat generated in these large furnaces is expected considerably to exceed anything met with hitherto, special features have been introduced to deal with it and prevent local overheating of the ore, which might lead to

slagging of the charge. The furnaces have an outside diameter of 20 ft., and the height from the topmost shelf to the ground is 50 ft. The weight of the centre shaft alone is over 30 tons. Nevertheless it can be revolved with one hand without effort. The rabbling arms are of special construction, and so fitted into their sockets on the shaft that the supply of cooling air to each arm can be independently controlled. The capacity of each furnace is between 35 and 40 tons of 48 per cent. pyrites per day, and correspondingly higher on ores with lower sulphur contents.

So satisfactory have the developments proved in practice that an even larger machine is being built having an outside diameter of 21 ft. 6 in. and containing the features described above.

It is not only in the large-size apparatus that the firm have progressed, but they have supplied something of great importance for experimental work—namely, small roasting furnaces only 6 ft. diameter, but giving a roasting area of 60 sq. ft. This small type experimental apparatus must find numerous applications in connection with new developments.

This firm have been well known for their screening plant and their vibrating "Overstrom" screen of steel construction, and the elimination of the timber frame has proved to be a very great advance, as it makes a very robust apparatus, less clumsy to handle.

In order to meet the increased demand for coke screening, they have developed a new type which is being built in lengths of over 20 ft. and fitted with meshes in series in such a way that either the largest or smallest sizes can be taken out first. This reduces to a minimum the amount of breeze formed by abrasion and crushing during the process of screening. It is well known that the grinding of coke is one of the serious disadvantages of grading coke in machines of the revolving drum type.

### Hadfields, Ltd.

The development of special steels with very high heat-resisting qualities by Hadfields, Ltd., has been exceptionally successful, and during the past year their heat-resisting products have been used for such very difficult conditions as the revolving arms or rakes in calcining furnaces of the tray type. The conditions under which these have been applied, where temperatures of around 900° C. are carried without any difficulty, and without cooling, either by air or water, show clearly the extraordinary success that has crowned their efforts in this direction. It has been applied not only to this type of calciner, but also to the rotary tubular type where the conditions are extraordinarily difficult, and there is no doubt that before long it will be quite a practical proposal, with this class of metal, to have calcining furnaces operating with temperatures approaching 1300° C.

The above remarks must not convey the impression that it is in heat-resisting steels alone that Hadfields' new developments have been so successful. They are also making special corrosion-resistant steels for such materials as phosphoric acid and other highly corrosive chemicals.

### Whessoe Foundry and Engineering Co.

A development of very great importance in connection with the prevention of evaporation in the storage of benzols and petrol is being carried out by the Whessoe Foundry and Engineering Co. It is common knowledge that the loss from storage tanks is very high in this country, but in tropical countries the loss represents very serious dimensions. To obviate this a patent apparatus known as Wiggins Floating Roof is being developed by the Whessoe Foundry Co., which consists of a very ingenious floating roof which floats on the top of petrol and rises and falls with the height of the spirit. It is estimated that the loss in petrol under certain conditions with an ordinary cone roof exceeded 2,200 gallons in 90 days, whereas with this new type roof the loss was little more than 600 gallons. Certainly a development of this kind is well

worth consideration. Means are provided for draining the rainfall or water from melting snows from the roof itself, and altogether it is an interesting development.

This firm are also making some very interesting new plant for gas storage under pressure, and have a specially designed holder for supplies of gas at pressures up to 50 lb. per sq. ft. In view of the modern methods of pressure distribution that are coming into vogue more and more, this is a development that will be followed with very great interest.

#### **Broughton Copper Co.**

The Broughton Copper Co. have always been prominent in new work associated with their manufactures of non-ferrous tubes and rods, and have recently carried out some very useful work for chemical engineers in the production of their Duplex Tubes. In this type of work they can take a copper tube and either line it or cover it completely with aluminium. In the same way they can use tin as a lining or covering, or they can cover steel tubes inside or out with copper. In fact, almost all of the non-ferrous alloys can be used as a covering or a lining, and seamless drawn tubes can be supplied in diameters from  $\frac{3}{4}$  in. right up to 9 in. and 10 in.

They have been developing further their aluminium bronze, which contains no zinc, and are now supplying sheets of this alloy, as this type of bronze can often be used where copper is found to be unsuitable.

The Broughton Copper Co. have an excellent technical staff to deal with all these special features and are considered to be well ahead of continental rivals in their specialities.

#### **Brown, Bayley's Steel Works**

Brown, Bayley's Steel Works, Ltd., have always been known for their enterprise in acid-resisting alloys that are particularly useful for chemical engineering work. One of the most notable features of this year's work has been the development of the use of low-carbon material containing 16 to 18 per cent. chromium. Material of this type—really a high chromium stainless iron—has been known for some time to have adequate resistance to the attack of many chemicals, and very large quantities of it have been used in U.S.A. for the construction of nitric acid plant. Its use in this country, however, did not find favour owing to its very low impact value. Chemical engineers here have in the past looked askance, and rightly so, at a material that appeared to be intrinsically brittle.

This disability, however, has been completely overcome by a special form of this material which is very tough and ductile. In the form of bars and plates it will give, for example, after suitable heat treatment, a tensile strength of 30/45 tons per square inch, combined with an Izod impact value of 40 to 60 ft. lb., a vast improvement on the 3 or 5 ft. lb. which was the usual value obtainable in the past from such high chromium material.

Stainless iron of this type has great advantages from the point of view of plant construction. It can be handled in a boilermaker's shop with almost the same ease as ordinary boiler plate steel. The bending, flanging and forming of plates, riveting, and the caulking of joints are easily carried out. The ease of manipulation in these respects is due to the fact that, unlike the austenitic steels, for example, its hardness does not increase very rapidly when it is cold worked. Already many tons of this material have been used for the construction of chemical plant, particularly for nitric acid, to which corrosive chemical it possesses the same resistance to attack as the more expensive austenitic chromium-nickel steels.

As regards the austenitic materials, the use of "Anka" steel has steadily increased. As a result of its high nickel content, "Anka" steel resists a wider range of chemicals than does the straight chromium iron. Owing to its particular composition, "Anka" steel is more amenable to boiler shop processes than many other somewhat similar types of steel and on this account is widely specified for plant construction. Notable features in connection with its use have been the development of modified types ("Anka 11" or "B.B. 4.K" and "Anka 111") which resist a still wider range of chemicals than the parent type, due to their content of other metals besides chromium and nickel. For example, their resistance to sulphuric acid of certain concentrations is very much greater than that of ordinary "Anka."

The number of purposes for which these special steels are required is bound to increase as their properties become known. A certain amount of knowledge is necessary in connection with the heat-treatment of these special steels for ordinary fabrication in the boiler shops.

#### **Hydronyl Syndicate**

The Hydronyl Syndicate, Ltd. make a speciality of the Lessing Patent Contact Rings, and although they have not made any variation in the forms of the rings themselves, they have made very great strides in increasing their application, which is really the important side of the business.

#### **Macinlop**

Macinlop, Ltd. have been pursuing their rubber investigations on a line which is very interesting to chemical engineers, namely, the rubber lining of large tanks in which to carry chemical products in bulk on tank wagons. This development has been carried to such a state of perfection that the old method of carrying by carboys has been, in a great many instances, entirely displaced by the bulk system, using large rubber lined tanks. In this way the acids can be conveyed direct from factory to factory either by rail, road or canal.

#### **Cannon Iron Foundries**

The Cannon Iron Foundries, Ltd. are increasing more and more their use of enamelled ironware, their special grey acid-resisting enamel being one of the most robust forms of enamelling that can be used in chemical works. During the last year they have specialised in a new design of mixing pans lined with their grey acid-resisting enamel. They make a speciality of interchangeability in the various parts, so that when breakages take place new parts can be ordered without causing delay.

#### **Thompson Bros. (Bilston)**

There has been a great change in late years in the construction of chemical plant, due to the use of welding as against riveting. Welding, both by acetylene and by electricity, is common enough, but both methods have suffered because so much work was done by people who did not really understand the technique of welding. Thompson Bros. (Bilston), Ltd. have not only perfected their system of welding, but they have extended their knowledge to the more difficult problem of making perfect welding for stainless steel. They now claim that there is practically freedom from inclusion of slag, gases, etc., in the weld. The mechanical strength is much greater, so that a guarantee can be given in the majority of cases that the weld is as resistant in a given medium as the plate itself. Not only so, but the weld is definitely ductile and can be worked after the weld is accomplished. Anyone who is interested in chemical plant construction knows how important these points are.

#### **Prodorite**

The necessity for the simple construction of tanks in acid-resisting material is one of great importance, and Prodorite, Ltd. show a great advance in this connection. Their method of lining tanks with the Prodorite material has been known for some time and very successfully used, but in the last year they have constructed sectional tanks of their material, which can be built up to any reasonable height. The method of construction is to have a baseplate with an arrangement of sockets and into this a Prodorite ring is dropped. These rings are 3 or 4 feet high and are interchangeable, so that the rings can be superimposed upon each other until the height of tank required is built up. The construction is simple, and for the bulk storage of hydrochloric acid has a great many points to recommend it. It has obvious advantages over the use of Yorkshire stone. The company are still going on with their special linings for existing tanks.

#### **Edgar Allen**

The expression "Stainless Steel" is becoming a name that may cover a multitude of sins or virtues, and in order to show that there is "stainless steel" and "stainless steel," the product that the Edgar Allen firm have developed is called by them "Maxilvry." This "Maxilvry" steel is made in billets, forgings, bars, and, in fact, any of the usual forms. It is malleable, can be satisfactorily welded, and is, in fact, a very important product for use in chemical engineering plant. No stainless steel is a cure for all ills, and it is advisable in using any steel of this kind to get the advice of the maker

to determine what exactly are its uses and the acids or alkalis it will resist. In order to help users Messrs. Edgar Allen have produced a very useful booklet which describes their product.

#### Kestner Evaporator and Engineering Co.

The drying of chemical products has always been of very great importance to the industry, and an apparatus that can be used not only to dry the product, but to leave it in a powdered form when finished, is of great interest. The Kestner Evaporator and Engineering Co., Ltd. have patented a spray dryer in which the drying is carried out by means of hot air, and in making this apparatus during the past year air heaters have been developed, taking advantage of the latest developments of high temperature steel. Much important work has been carried out in testing suitable materials for this purpose, and the company have designed and built air heaters of this special construction.

While this company are still developing their well known forms of plant, they have been making very material progress in the investigation and use of oil for heating purposes instead of steam or gas. The heating of this circulating oil by means of electricity is a distinct line of progress that has been very successful and seems to have a big future before it, the more so as electric current is not only obtainable over a wider area but at much lower prices.

#### Oxley Engineering Co.

The homogeneous lead-coating of mild steel and other metals is always an important branch of chemical engineering. This is an industry that had been carried on by Kestner's for many years, and their process has been taken over by the Oxley Engineering Co., Ltd. A very large and complicated amount of chemical plant has been constructed, all homogeneously lead covered. This process has been in use since the early days of the war. It is an English process entirely, and plant that was delivered during the war period constructed in this way is still in operation.

#### Thomas Broadbent and Sons

The filtration of chemical products is always one of the problems of importance in various chemical processes, and new forms of continuous filtration are always being looked for in the chemical industry.

Thomas Broadbent and Sons, Ltd., whose name is so well known in connection with centrifugal machines, claim that they have made exceptional progress in the last year with their solid basket type centrifugal, so that it can now take the place of the filter press in a large number of filter processes. The working is very simple as, instead of using a perforated basket with different forms of cloths, they use a non-perforated basket, filling the liquor in such a way that when charged centrifugal action drives the solids in a mass against the side, and a very simple form of adjustable scoop rapidly removes the surplus water or liquid and thus the material is de-watered.

This type of apparatus seems to have met with considerable success in a number of industries, where its application has been found suitable.

#### Thomas Firth and Sons

The advances made in chrome steel of the well-known Staybrite brand are of very considerable interest, particularly to the food industry. In special steels of this type, it is not enough simply to produce the steel itself. Thomas Firth and Sons, Ltd. have taken advantage of their exceptionally well-equipped research laboratories to help plant manufacturers to develop the technique of manufacture. The technique of manufacture in steel of this kind is important, because it adapts the product for work for which it could otherwise not be used.

Firths are not manufacturers themselves of chemical plant, but this technical organisation of theirs is available both for the user and for the manufacturer. Consequently they have a wealth of experience available, whereby problems that look insoluble have not only been solved, but have created new advances in the industry.

#### Baker Perkins

In the use of mixing and kneading machines Baker Perkins Ltd. are known by chemical manufacturers throughout the world, and the newer industries, such as the silk industry and the electrical cell industry, are finding them ready to meet the special conditions now demanded.

Their new system of machines, which are jacketed so as to take very low temperatures during their mixing operations or alternatively very high temperatures, and which they have developed in the last year, have proved most successful. Very many processes now require reactions to take place at very low temperatures and others at equally high temperatures. In the high temperature mixing it is possible to obtain temperatures in the jackets of about 300° C., which corresponds to steam pressures of 1400 to 1500 lb. per sq. ft. However, this question of pressure is overcome by utilising a patent oil circulation system, so that, although the high temperature is obtained, the pressures are actually very low.

For low temperatures specially constructed vessels are used; and by means of their special construction they take care of the high degree of expansion and contraction of the metals found under these conditions.

Not only in this direction, but in hydraulic accumulators, this firm have made very important advances, for instead of utilising the well known weights that are commonly employed, they have a very interesting and useful method by which air pressure takes the place of the weights. It can be adopted both in the largest and the smallest sizes and for pressures up to 1½ tons per sq. ft.

#### Chemical Engineering Group

UNDER the chairmanship of Mr. H. Talbot, the Chemical Engineering Group of the Society of Chemical Industry has, during the past year, continued its policy of presenting useful papers for discussion, either at its own meetings or in conjunction with local sections of the Society, as opportunity arose. In the latter connection, joint meetings have been held at Birmingham, Nottingham and Bristol, and the following works, etc., visited: Henry Wiggin and Co., Birmingham; the University of Nottingham and Manlove, Alliott and Co., Ltd., Nottingham; the National Smelting Co., Ltd., Avonmouth.

Prominent among the papers read in London is one read in January by Professor B. P. Haigh on "The Relative Safeties of Mild and High-Tensile Alloyed Steels under Alternating and Pulsating Stresses," and one read in October by Mr. J. Davidson Pratt, general manager of the Association of British Chemical Manufacturers, on "Rationalisation—its Meaning and Application, with special reference to the Chemical Industry." Each of these papers proved a particularly good exposition of its particular subject, and the first one—by Professor Haigh—has since achieved the much wider publicity the importance of its subject undoubtedly deserved by being read before other scientific bodies. In February, Professor W. E. Gibbs presented an important paper on "The Role of Surface Energy in Chemical Engineering." Papers read at the joint meetings with local sections included a paper on "The Design and Operation of Gas-Heated Furnaces," by Dr. C. M. Walter (read in Birmingham); "The Scientific Heating of Liquids and Gases," by Mr. J. Arthur Reavell (read in Nottingham); and "The Treatment of Flotation Concentrates Preparatory to Zinc Smelting," by Mr. Stanley Robson (read in Bristol).

At the annual general meeting, held in May, at which Dr. H. Levinstein, President-elect of the Society, was the principal guest, the chairman initiated a discussion on "How can the Chemical Engineering Group best assist in the development of the science and practice of chemical engineering?" and a lively debate ensued, in which all phases of the Group's past, present and potential activities were reviewed.

Vacancies on the committee which had occurred during the year and under the operation of the rules were filled at the annual general meeting by the election of Dr. S. G. Barker, and Messrs. C. Barber, H. W. Cremer, I. P. Llewellyn, F. M. Potter, S. Robson and S. J. Tungray. At the same time, Mr. H. J. Pooley was re-elected honorary secretary, which post he resigned on his appointment as general secretary of the Society of Chemical Industry. Mr. W. F. Darke, of Bristol, was appointed in his place.

Some important joint meetings are in prospect for early in 1930, viz., February 7, when at a joint meeting with the Birmingham Section the subject of "Electric Furnaces" will be discussed, and on March 7, when the question of the "Insulation of Heated and Cooled Surfaces" will be considered jointly with the Glasgow Section.

## Conditions in the Heavy Chemical Industry in 1929

By P. Parrish, A.I.C., M.I.Chem.E.

*That there can be nothing stereotyped about the heavy chemical industry is becoming increasingly evident. Many factors are operating which render it more difficult each year to predict whether markets are likely to be retained, held with difficulty at a sacrifice of revenue, or definitely lost. Nations are revealing increasingly a desire to become self-supporting. This is particularly the case with the production of heavy chemicals and fertilisers. In the following article, Mr. Parrish reviews the tendencies that have revealed themselves during the year.*

THE growth of the synthetic ammonia industry in Belgium, Holland, Italy, Poland and Japan, not to mention more important European countries and America, and the known projects which concern Australia, Chile and China, are unchallengeable testimony to the course of development of this industry. Amalgamations appear to be the order of the day, and yet they may or may not represent rationalisation. Most of the important European countries now have an exceptionally large organisation actively concerned with the heavy chemical industry. The I.G. (Germany) led the way, and was followed by I.C.I. (Great Britain), Kuhlmann (France), Montecatini (Italy) and l'Union Chimique Belge (Belgium).

It is, however, being realised that the size of an organisation in itself may be a doubtful advantage unless the larger resources that it provides are well employed. On the other hand, the elimination of wasteful competition is obviously good, cheapening production as it does to the benefit of the consumer.

### Productive Capacity for Synthetic Ammonia

The danger with the synthetic ammonia industry, regarding it in its widest sense, is that productive capacity is likely to outrun purchasing power, with a possible resort to keen-cut competition, and any savings in production may well be lost in expensive sales propaganda. The sequel will doubtless be international ententes and the allocation of quotas, pending an increase of the world's population and a consequent increased demand.

The Mont Ceniz process, which has already been worked on a large scale near Dortmund, offers important advantages over other methods. Far lower temperatures and pressures are employed, and lower capital costs are involved. It is a process that can be put into operation more speedily than any other, and profits can be earned within eighteen months of the erection of the plant.

Some interesting technical data concerning the production of synthetic ammonia at Ostend, representing the important work now being carried out by the Union Chimique Belge, have been published. The cost of hydrogen is an outstanding item in the cost of synthetic ammonia. Coke oven gas is generally admitted to offer the cheapest form of hydrogen. In Germany, previous efforts at the purification of coke oven gas before separation of hydrogen consisted of removing sulphur with iron oxide, followed by compression and washing under pressure with water to remove  $\text{CO}_2$ , a final decarbonation with caustic soda, and drying under pressure with calcium chloride. The treatment at Ostend has been modified, and is said to produce a hydrogen purer than any other method using a non-electrolytic gas.

### Fixation of Nitrogen by Micro-organisms

Although work is proceeding on the fixation of nitrogen by micro-organisms, little further information has been published. How far such a notoriously slow process is likely to challenge existing synthetic methods remains to be seen. Probably by the time sufficient is known of the vagaries of azo-bacteria and legume-bacteria, improved methods will have been introduced into synthetic ammonia factories, which will ensure a continued lease of life.

### Scientific Utilisation of Coal

The prosperity of Great Britain is centred in the reorganisation of the coal industry and the more scientific utilisation of coal. The iron and steel industries need cheap fuel and improved technique. It is known that several collieries could be restarted, and reasonably profitable results obtained, if coal were carbonised, oven coke produced and the hydrogen of the coke oven gas utilised for the manufacture of synthetic ammonia or for hydrogenation processes. It is in this direction that colliery companies may be compelled to turn. What has been done in France and Belgium is equally capable of achievement by this country. The one difficulty is over-

production, reference to which has already been made from the viewpoint of the world. The immediate consideration is more circumscribed, involving as it does Great Britain's outlook.

What was remarked two years ago, to the effect that the future of our Empire is the future of Empire trade, is no less true to-day than it was then; indeed, the lapse of time has merely strengthened the soundness of the view. There is an unlimited field for the expansion of our Empire trade, and it is here that our efforts should be concentrated, in the hope that suitable outlets can be found for our heavy chemical products.

A glance at the patent literature of the year shows that the hydrogenation of coal, coal liquefaction, and the distilling and cracking of coal oil and low-temperature tars have been receiving earnest attention. While no one doubts that low-temperature processes are sound as regards the abatement of the smoke nuisance, the growing importance of some satisfactory method for treating coal oil and low-temperature tar as a factor in the economy of the process scarcely needs emphasis.

### Coal Liquefaction

Dr. Lander, in his Cantor lecture this year, indicated that the experiments, both at the Fuel Research Station and elsewhere, had shown that 110 to 130 gallons of distillable oil per ton of coal actually used could be obtained. A problem presenting some difficulty was the introduction of a solid material such as coal into a vessel containing hydrogen under a pressure of 200 atmospheres. Bergius had solved this by mixing ground coal with a liquid organic vehicle so as to form a paste which could be pumped by special appliances. Iron oxide serves as a catalyst, or as a means of removing hydrogen sulphide from the gas evolved. The chief value of the fractions from the hydrogenation of coal would be either as a motor spirit or as fuel oil. Indications are not wanting that certain of the fractions may be of value as lubricating bases.

Dr. G. T. Morgan has studied low-temperature tar, and the results of his investigations indicate that by the solvent method an extraction of about 1.6 per cent. of wax was obtainable. It was readily bleached, and of comparatively high melting point. Higher aromatic hydrocarbons amounted to more than 1 per cent. Low-temperature tars contain a relatively high percentage of phenols, which can be freed from the tendency to redden on exposure. These, and their chlorinated derivatives, were germicidally very active. When condensed with formaldehyde the phenols yield both fusible and infusible resins. The former were applicable as lacquers and varnishes, and the latter, when mixed with fillers, could be used in making moulded articles, capable of being machined.

### American Association with the I.G.

As illustrating the activity of America in association with the I.G., mention must be made of the increased production of petrol by the Standard Oil Company of N.J. during this year. The experimental plant erected for the treatment of distillation and cracking residues, and asphaltic and high-sulphur oils, according to I.G. methods, has been brought successfully into action, and by the end of this year it was anticipated that a full-scale plant would be in operation.

It is known that rapid development of the cracking process and the increasingly large proportion of oils of high-sulphur content which are used for cracking stocks have introduced critical problems into oil refining. After a careful study of this problem, it was concluded that a lining made of non-metallic materials, of ceramic or mineral-like character, would be the most economical and satisfactory solution. A paper by Morrell and Faragher, in *Chemical and Metallurgical Engineering* (October, 1929), is peculiarly interesting in this connection.

What has emerged during the last twelve months more clearly than ever is that improved technique in almost every

phase of sulphuric acid manufacture is to be anticipated. The quantity of brimstone used in Great Britain will sensibly diminish during the next year, and an increasing quantity of pyrites will be burned. The use of Rio Tinto washed pyrites fines is likely to grow apace.

The equipment and operation of mechanical pyrites burners can be improved considerably. Hitherto the variations of speed have been by definite gradations. Thus their operation has been insufficiently flexible, and their performance with pyrites of different characteristics can scarcely have been accurately or adequately determined. Infinitely variable gears are now made in a compact form, and can, at moderate expense, be used to afford wonderful elasticity as regards speed of operation. It is certain that optimum throughput of many of these types of mechanical burners depends upon

acid by the process in question. The size of the unit affects the capital expenditure very sensibly. The following figures are given as representing the conditions in the United States.

CAPITAL EXPENDITURE ON CONTACT PLANTS IN THE UNITED STATES.

Size of Unit.	Sulphur Material.	Capital per Ton of 66° Bé Acid.	Buildings.
15,000 tons per year of 66° Bé acid .....	Brimstone	\$12.25*	Not included
13,000 tons per year of 66° Bé acid .....	Pyrites	\$18.55*	Not included
17,500 tons per year of 66° Bé acid .....	Brimstone	\$15.70	Including Buildings

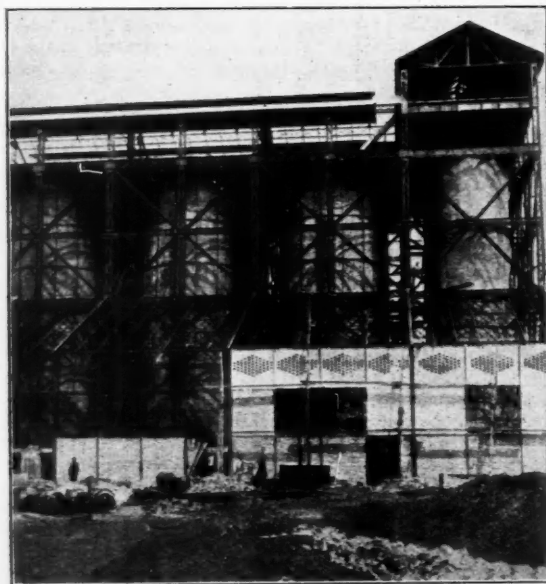
\* N.B.—At least 20 per cent. should be added to the above figures to cover the cost of buildings.

A chamber plant in the United States, capable of producing the equivalent of 23,000 tons of 66° Bé. acid, using brimstone as raw material, can be erected for \$14 per ton, including buildings.

It must, however, be remembered that a large modern plant on the Gaillard liquid phase system, such as is represented by the photographs can be erected in the United States to-day, including rotary mechanical sulphur burners, for \$8.75 to \$9.00 per ton of 60° Bé. acid. In face of such figures it is doubtful whether the contact process will rapidly displace the chamber system, particularly as improved methods are being introduced which afford an increased life to the last-named.

#### The Gaillard Liquid Phase System

The Gaillard liquid phase system of sulphuric acid manufacture continues to extend. American works have shown a keen interest in this method of manufacture, which is becoming increasingly popular. Over four hundred turbo-dispersers are in operation in all parts of the world. The projection of acid on the internal walls dislodges the gas film, which is perhaps the most insistent insulating medium. Thus the cooling effect by the latter system is alike direct and positive—a feature of outstanding importance. The idea of dispersing acid of varying nitrosities has not been developed, because no lining sufficiently inexpensive has been found. In any case, a lining (a) retards conduction of heat, and reduces to a minimum the advantage derived from the cooling influence of the acid on the walls; and (b) all indications point to a shorter life of the lead under these conditions.



GAILLARD LIQUID PHASE SULPHURIC ACID PLANT.

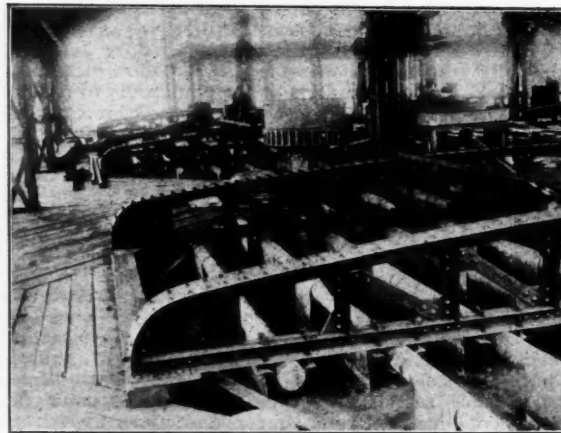
the selection of a specific speed of rotation for a given quality of ore. Apart from the delicacy of adjustment in the matter of speed of rotation, increased flexibility can be afforded by using air for cooling and by providing cooling arms, in addition to the rabbling arms, in those beds where the highest temperatures obtain. Not infrequently the fusion temperature of the ore is reached and slagging occurs. This could be avoided if cooling arms were provided on certain of the top beds.

There is reason to believe that the various types of static dust chambers which have hitherto been used in connection with mechanical pyrites burners will be dispensed with gradually, and electrostatic precipitation resorted to. The efficacy of the latter method is beyond dispute. The improved acid production which follows, no less than the saving effected in nitrate of soda consumption, more than justifies the installation of this type of plant. New forms of burners are likely to be developed, which may sensibly reduce the quantity of dust created. Experiments are proceeding in one or two directions and these are likely to be fruitful of improved results.

#### Sulphuric Acid Catalysts

The introduction of new catalysts of the vanadium type exploited by the Graesser-Monsanto and Selden companies, for the production of sulphuric acid by the contact process, now definitely challenges the chamber acid or nitration process. It is known that 10,000 litres of vanadium catalyst is necessary for the production of 75 short tons of  $H_2SO_4$  per day, that a litre contains 1.74 lb. of  $V_2O_5$ , and that the cost of the catalyst is about \$1.10 per litre. Apart from this, a royalty has to be paid per ton of sulphuric acid produced by the process for a period of years.

It is, however, difficult to obtain any reliable figures as to capital cost of plants, or cost of production of 100 per cent.



METHOD OF SUPPORTING THE TOP OF A GAILLARD SULPHURIC ACID PLANT—TURBO DISPERSER NOT VISIBLE.

With an unlined Gaillard tower chamber, in which chamber acid which is substantially free from oxides of nitrogen is dispersed, the probabilities are that the life of the lead will be at least twenty years. Evidence of this is shown by the freedom of the drips from turbidity, and the negligible depreciation of weight of samples of lead after several years' operation.

Improved methods for the dearsenication of sulphuric acid are called for, and these should involve granulation of the

arsenic sulphide so as to facilitate its handling and its profitable disposal.

#### Sulphuric Acid from Gypsum

A method for the manufacture of sulphuric acid from gypsum is due to I.C.I. and K. Gordon (E.P. 309,298); it takes advantage of the chamber process. The calcium sulphate is mixed with carbonaceous and siliceous materials in the usual way, and the mixture fired in a kiln. Excess air is admitted so that the effluent gases contain about 2 per cent. of oxygen and reach a temperature of 400 to 500° C. Additional air is then added in order to bring the oxygen content up to 8 or 9 per cent., in which condition the gases are suitable for Glover tower reaction.

In an article dealing with the significance of the production of sulphuric acid from anhydrite, it has been shown that the price of spent oxide is not likely to suffer adversely by reason of the developments at Billingham. On the contrary, the increasing difficulty of obtaining supplies of Sicilian sulphur must inevitably exert a reflex action, and the realisation for the sulphur content of spent oxide is likely to increase rather than decrease within the next few months.

#### By-Product Ammonia

The situation as regards by-product ammonia is as acute as ever. Despite the existence of an Ammonia Committee of the Institution of Gas Engineers, which has worked with commendable energy in the investigation of ammonia processes and suggestions, no definite solution of this problem has yet been found. It is true that one process has been suggested by which the strength of gas liquor can be increased appreciably at comparatively small expense. This is a process that can advantageously be employed, but it can only be a partial solution of the problem at best. It is no exaggeration to say that an effective solution has yet to be found. That such a solution exists appears certain, but it involves the adoption of new methods and a change of policy.

Meanwhile, new processes are being introduced. Two such are the Tern Elektrostikstoff and the Koppers C.A.S. The former is based on the combustion of spent oxide and the conversion of the resultant  $\text{SO}_2$  to  $\text{SO}_3$  by the use of the electric arc. The sulphuric anhydride is brought into contact with ammonia and sufficient water, arising from the distillation of gas liquor in a special way, in an electrostatic precipitator, and sulphate of ammonia is deposited. By this method it is claimed that the final product, known as Elektroammon, can be produced for 90 to 100 Reichsmarks per ton less than ammonium sulphate, as hitherto prepared in the gas industry.

#### Elektroammon

But is Elektroammon in a suitable form for sale? Electrostatic precipitation invariably results in the production of a fine powder, which in the case of sulphate of ammonia would be susceptible to rapid caking. Large crystals of reasonably uniform size are now demanded.

Moreover, the treatment of sulphur dioxide by the electric arc must result in the formation of a mixture of  $\text{SO}_2$  and  $\text{SO}_3$ , which leads one to anticipate that Elektroammon consists of a mixture of ammonium sulphite and ammonium sulphate, which again introduces all the old difficulties associated with Burkheiser salt.

As regards the Koppers C.A.S. process, this is far too complicated for application to the majority of gasworks and coke ovens in this country, and certainly affords no advantage in point of cost over the existing dry method of purification for the recovery of sulphur. Moreover, the conversion of ammonium thiosulphate to ammonium polythionates by treatment with  $\text{SO}_2$  is not without difficulty, and the autoclavic reaction by which ammonium polythionates are converted to ammonium sulphate and sulphur has been a source of trouble with another process, where this reaction has been worked on a large scale. The two foregoing processes are not likely to constitute an immediate solution of the by-product ammonia problem, which, after all, is what is required.

A further report has been published by the Effluent Committee of the Institution of Gas Engineers. Attention is now being directed to the extraction of phenols from gas liquor as the most economical method by which the toxicity of spent effluent liquor can be reduced. Electrostatic precipitation of tar has led to unsuspected complications, and it is doubtful

whether this method will finally constitute the most economical one. It is still believed that the problem needs to be attacked from another angle.

#### Superphosphate Industry

The improved position which was evident from the reports of several superphosphate companies has been largely maintained. There is a tendency for calcium superphosphate of higher grades to be manufactured, although the superphosphate industry generally is of opinion that mono-ammonium phosphate or di-ammonium phosphate, or any of the concentrated fertilisers, is not likely to meet with immediate success. The question of the distribution of concentrated fertilisers is one that presents difficulties, and certainly needs to be carefully investigated. It is believed that the superphosphate industry is in a unique position for developing in certain directions which have hitherto remained somewhat obscure. Generally speaking, the fact that these works produce cheap acid lends increasing support to this view.

Why should the prosperity of the *Erste Nederlandsche Coöperatieve Kunststufabriek te Vlaardingen*, for the year ended June 30, 1929, be so marked in contrast with our own superphosphate works? The gross profit was 481,181 florins. The scale of operations is undoubtedly a factor, as also are the wonderful loading and discharging facilities provided. It is remarked in the report of this company that a gradual improvement in the superphosphate market was noticeable, and in the spring superphosphate was short in supply in Holland, and a higher price was commanded. The sulphuric acid plant operated to capacity, about 190,000 tons of acid being made. Vlaardingen is to-day the third port of Holland, with a shipping tonnage three-quarters that of Amsterdam.

#### Production of Concentrated Phosphoric Acid

Several modifications of the recognised process of manufacturing phosphoric acid by the interaction of calcium phosphate and sulphuric acid have been directed to producing the acid in a more concentrated form. A recent patent of the I.G., No. 318,116, protects the use of concentrated sulphuric acid for treating the phosphate, the mixture of sulphuric and phosphoric acids being used for treating further quantities of phosphate until the sulphuric acid content is exhausted. A further patent by the same company, No. 309,604, covers the use of calcium sulphate obtained from the above process, for the manufacture of cement and sulphuric acid.

The problem of disposing of calcium carbonate resulting from the anhydrite process has led to a patent (E.P. 305,760) for the manufacture of a fertiliser by mixture with ammonium nitrate. This is added in the form of a 90 per cent. solution, and the mixture is afterwards dried in a vacuum evaporator. It can be imagined that where the calcium carbonate can be utilised in this way freedom from ammonium sulphate is not so important, and the washing process may be somewhat simplified.

Whether 1930 will witness fresh developments in technique on the lines foreshadowed remains to be seen. Certain it is that the heavy chemical industry will need to be in the vanguard if it is to maintain unimpaired the prosperity which it has enjoyed during the last two years.

#### Harrison Memorial Prize Award

THE Harrison Memorial Prize Selection Committee, consisting of the President of the Chemical Society (Professor J. F. Thorpe, D.Sc., F.R.S.), the Institute of Chemistry of Great Britain and Ireland (Professor Arthur Smithells, D.Sc., F.R.S.), the Society of Chemical Industry (Dr. Herbert Levinstein), and the Pharmaceutical Society (Mr. L. Moreton Parry) has awarded the Harrison Memorial Prize for 1929 to Dr. Reginald Patrick Linstead.

The prize is given for conspicuously meritorious work in any branch of chemistry, pure or applied, and is to be regarded as an exceptional distinction to commemorate an exceptional man and to be conferred upon the young chemist under 30 years of age who, in the opinion of those best qualified to judge, has made a notable addition to our knowledge of chemistry.

The presentation of the prize will be made at the annual general meeting of the Chemical Society, Burlington House, Piccadilly, W.1, on Thursday, March 27, 1930, at 4 p.m.

## A Review of the Year's Chemical Inventions

By Our Patents Correspondent

*Attention is again drawn, in reviewing the chemical inventions of the year, to the very large proportion of British patents taken out by German interests, and to the fact that the great majority of the total number of chemical inventions come from large industrial corporations with considerable facilities for research.*

THE whole of the post-war period has been, for most industries, one of steadily increasing activity of invention. Some three or four years ago, however, it became evident that the industries based on pure and applied chemistry were beginning to occupy a unique position in this respect. The stream of chemical invention, as measured by the number of applications for patents in this country, rose rapidly to the proportions of a flood. That flood has advanced to a still higher level during the present year. Thus the I.G. Farbenindustrie Akt.-Ges. made during the third quarter of 1929 326 applications, as compared with 284 during the corresponding quarter of 1928, and the proportional increase is even higher in the case of Imperial Chemical Industries, Ltd.—viz., 59, as compared with 32 applications, during the quarters referred to.

### Activities of Large Corporations

Most of the current inventions in the industry emanate from the large industrial corporations, such as these, with their elaborate facilities for research. Indeed the proportion not so emanating is probably smaller than in any other field, the electrical industry not excepted. The present phenomenal activity is notable in all branches of pure and applied chemistry, metallurgy, and chemical engineering. If there is any well-defined field which is at present of outstanding fertility it is probably the rapidly developing artificial silk industry. Thus, a leading British firm in this branch made 53 applications during the third quarter of 1929, as compared with 36 during the corresponding quarter of 1928.

Any selection of inventions for special notice in a brief review is necessarily defective in view of the enormous field covered, but the following notes give some indications of the main trends of invention in a few of the leading branches of chemical industry during the year. The numbers of the specifications referred to are given in parenthesis, together with the volume and page of THE CHEMICAL AGE where an abstract of the specification may be found.

### Treatment of Coal

The fundamental importance in the present-day industrial system of the economic utilisation of coal and other carbonaceous raw materials is reflected in the large number of inventions in this field. A considerable proportion of these inventions relates to the hydrogenation of coal and oils, usually to obtain products suitable for use as motor fuels. Thus a mixture of coal and oil may be subjected to an electric arc or spark at 300° C. under a pressure of 150 atmospheres in presence of hydrogenation catalysts (301,949, Vol. XX, 52). Treatment with excess of hydrogen at above 550° C. and above 20 atmospheres is employed for converting non-aromatic hydrocarbons present into aromatic, unsaturated aliphatic, and hydroaromatic hydrocarbons boiling below 200° C. (302,253, Vol. XX, 81). A preliminary treatment of the catalyst at high temperatures with certain gases has been found to be advantageous (311,251, Vol. XX, 593). The use of nascent hydrogen derived from water has been described (313,963, Vol. XXI, 178).

Much attention is also being directed to defining the best working conditions in the known hydrogenation processes, and to improving the plant—particularly, in the latter case, the construction of apparatus adapted to withstand the high temperatures and pressures used and to facilitate the separation of the gaseous and liquid products. Improvements in catalysts employed in hydrogenation processes applied not only to the raw materials, coal, oil, etc., but also to various classes of organic compounds, have also been numerous.

### Removal of Sulphuretted Hydrogen

Processes for the removal of sulphuretted hydrogen and ammonia from coal gas include the use of an iron thiosulphate solution (299,765, Vol. XX, 13), and the use of a liquor containing oxysulphur compounds of iron and iron hydroxide in such proportions that the scrubbing is complete in a single operation (309,116, Vol. XX, 547), the spent liquor in each case being regenerated by means of sulphurous acid. Pro-

cesses for removing sulphuretted hydrogen and organic sulphur compounds include passing the gas over various heated metals or oxides associated with metals or oxides of the sixth or seventh group (305,026-7, Vol. XX, 340), or over certain metals or oxides on an inert carrier, the proportion of metal or oxide to carrier being such that the composition cannot retain more than 10 per cent. by weight of sulphur, so that when the composition is regenerated by air or oxygen the heat liberated does not damage the activity of the metal or oxide (309,585, Vol. XX, 595), or over the usual heated metals to which about 5 per cent. of lead, bismuth, or copper has been added (319,396, Vol. XXI, 387).

Several inventions for obtaining mixtures of hydrogen and carbon monoxide by treatment of coal gas, hydrocarbons, or solid fuels have been noted.

### Hydrocarbons

The conversion of hydrocarbons into mixtures of hydrogen and carbon monoxide has been effected by passing them, with water vapour or carbon dioxide, with or without air, over catalysts arranged in tubes of chromium-nickel alloys heated externally to 600—1,000° C. (301,969, Vol. XX, 52), and mixtures of methane and other hydrocarbons and steam have been converted into hydrogen and carbon dioxide by use of a catalyst comprising a precipitated nickel hydroxide reduced with hydrogen at 350° C. (314,944, Vol. XXI, 224). Various methods have also been described for effecting the reverse of these processes, viz., the production of liquid hydrocarbons from hydrogen and oxides of carbon, e.g., by heating the gaseous mixture under pressure in presence of catalysts such as a metal of the eighth group containing a small proportion of an alkali metal (312,717, Vol. XXI, 9), or heating it at 500° C. in presence of finely-divided iron, nickel, or cobalt, or sulphides of iron, molybdenum, or tungsten (312,916, Vol. XXI, 114).

Methane or ethane may replace some or all of the hydrogen in some processes of this type, and oxygenated products, e.g., acids, esters, alcohols, or ketones, are obtained in some cases. Various methods have been suggested for overcoming the main difficulty in these reactions, viz., the poisoning of the catalyst by deposition of carbon. Thus the catalyst has been maintained at a uniform temperature by means of a cooling medium (310,999, Vol. XX, 570), and the reaction has been effected under special conditions of circulation of the gases (313,467, Vol. XXI, 57) for this purpose.

### Miscellaneous Gas Reactions

A few inventions relating to the production of acetylene by subjecting gases containing methane and other hydrocarbons to high tension arc discharge are concerned mainly with the construction and disposition of the electrodes and the direction and velocity of the stream of gases in relation thereto.

An interesting invention concerned with the transformation of water gas, ethylene, acetylene, etc., into liquid hydrocarbons is based on the action on the gases of an intense beam of  $\beta$ -rays having a velocity above half the velocity of light (309,002, Vol. XX, 453).

Among recent polymerisation processes and syntheses may be noted the production of higher olefines and diolefines, such as butadiene, from ethylene and other lower olefines by the use of high temperature electric discharges (315,249, Vol. XXI, 155), the production of benzene and other aromatic hydrocarbons by heating methane to 1,000—1,100° C. for  $\frac{1}{16}$  to  $\frac{1}{8}$  seconds (316,126, Vol. XXI, 294), the production of higher hydrocarbons by heating mixtures of olefines and acetylene, with provision for preventing deposition of carbon by ensuring that the hot gases come into contact only with elements of the fourth group, viz., tin, silicon, lead, or carbon (graphite), or with zinc or aluminium (316,422, Vol. XXI, 202), and the production of liquid polymerisation products from methane by subjecting it to an alternating high tension electric field producing a silent electric discharge and having a frequency of 8,000—12,000 cycles per second (317,344, Vol. XXI, 265).

### Rubber

A revival of interest in the synthetic production of rubber-like products, particularly on the part of German chemists, is indicated by some recent inventions in this field. A few relate to the production of unsaturated hydrocarbons (isoprene, butadiene, etc.) suitable for use as starting materials. Such are, for example, the production of unsaturated from saturated aliphatic hydrocarbons by heating in the presence of active charcoal or of catalysts obtained by heating iron or zinc humate in absence of oxygen (301,402, Vol. XX, 12), or by heating to 500–800°C. in presence of oxygen-transferring catalysts and of gases containing oxygen or sulphur (305,603, Vol. XX, 258), and the production of butadiene by passing the vapour of 1:3-butyleneglycol over red phosphorus distributed over a dehydrating catalyst or over pumice or other dispersing agents (315,595, Vol. XXI, 156).

A process for obtaining the 1:3-butyleneglycol for this purpose, with methane as the starting material, has also been described (307,808, Vol. XX, 383). New methods of polymerising and coagulating the latex obtained by emulsification of the hydrocarbons have been numerous, and include cooling below 0°C., thawing, and separating the coagulum (304,207, Vol. XX, 283), the use of certain proteolytic enzymes (307,375, Vol. XX, 359), the use of albuminous substances as aids to polymerisation (312,949, Vol. XXI, 33; 319,801, Vol. XXI, 428), and the use of boron trifluoride with certain accelerating agents (313,067, Vol. XXI, 33). Polymerisation of butadiene by agitation in an autoclave with sodium hydride at 30–50°C. has also been suggested (315,356, Vol. XXI, 244). A process of another type for producing artificial rubber is one based on the treatment of petroleum with nitric acid or sodium peroxide in the presence of soda-lime or sodium acetate, distillation of the neutralised liquid, and heating portions of the distillate with glacial acetic acid or formaldehyde, together with raw rubber as catalyst. A syrupy substance is obtained from which an elastic vulcanisable product is precipitated by acetone or alcohol (312,741, Vol. XXI, 9).

### Vulcanisation

Among recent vulcanisation processes are one for incorporating selenium or selenium compounds in a colloidal state into natural or artificial rubber (311,372, Vol. XXI, 34), and one for incorporating similarly selenium-sulphur compounds in the form of a paste obtained by triturating the powder with oils or fats (315,554, Vol. XXI, 156).

Research relating to the acceleration of the vulcanisation process continues unabated, especially in Germany and the United States. Compounds used as accelerators include many mercaptothiazole derivatives (300,949, Vol. XX, 82; 302,142, Vol. XX, 159; 303,827, Vol. XX, 259; 308,275, Vol. XX, 508; 316,932, Vol. XXI, 337), guanidine derivatives (312,069, Vol. XXI, 90; 315,554, Vol. XXI, 156), alicyclic bases and their derivatives (300,208, Vol. XX, 53; 311,735, Vol. XXI, 58), reaction products of benzoynitrophenyl sulphur halides and alkali salts of organic sulphides (302,143, Vol. XX, 159), hydrolysed halogen-free derivatives of aldehyde-amine condensation products (302,176, Vol. XX, 159), and thiuram sulphide derivatives (303,535, Vol. XX, 129).

### Ageing and Anti-Oxidising Agents

Inventions relating to agents for improving the ageing qualities of rubber emanate almost exclusively from American research organisations. Among suitable substances specified in recent specifications are reaction products of aromatic amines and aliphatic acids or esters (302,144, Vol. XX, 159), non-accelerating substituted phenol derivatives (302,147, Vol. XX, 159), dinaphthylamines (302,271, Vol. XX, 189), certain phenol-amine condensation products (305,195, Vol. XX, 359), dihydroxydiaryl derivatives (305,647, Vol. XX, 384), arylaminobenzyl alcohols (307,013, Vol. XX, 454), thiodylarylamines (309,161, Vol. XX, 547), disubstituted *p*-phenylenediamines (312,630, Vol. XXI, 114), and anilidonaphthoquinones (313,486, Vol. XXI, 156). Resinous non-accelerating condensation products of aldehydes and secondary aromatic amines have been suggested as anti-oxidants in vulcanised rubber and as agents retarding resinification and tack in raw rubber (317,786, Vol. XXI, 388). A British invention (318,717, Vol. XXI, 337) comprises the use for preserving rubber latex of boron compounds, *e.g.*, sodium pentaborate or a mixture of borax and boric acid, imparting to the latex a pH value between 6 and 9.2.

The economic utilisation of raw phosphates continues to receive much attention. Apart from numerous improvements in the production of superphosphates and of nitrogen-phosphate mixed fertilisers, such processes may be mentioned as sintering with acid or basic materials and a strong reducing agent such as carbon or iron pyrites, so that the product contains no free alkali or alkaline earth bases and the sulphur is completely eliminated (300,961, Vol. XX, 82), calcination in two stages with alkali carbonates or magnesium compounds and silica in presence of steam to obtain a citrate-soluble product with elimination of fluorine (301,022, Vol. XX, 83), sintering with an alkali sulphate and a strong reducing agent such as carbon, iron pyrites, or calcium sulphide, to obtain in some cases a sulphur-free product (306,086, Vol. XX, 411), and various processes, mainly of Swedish origin, based on leaching with sulphuric acid or with soluble sulphates.

### Nitrate Fertilisers

As examples of recent developments in simple nitrate fertilisers, the following inventions are of interest:—A mixture comprising potassium nitrate and sodium nitrate in specified proportions (303,916, Vol. XX, 158), the fertilising value of such a mixture being found to be greater than that of a fertiliser containing either constituent alone representing the same amount of nitrogen; a mixture obtained by adding calcium carbonate (obtained as a by-product of the preparation of ammonium sulphate from ammonium carbonate and calcium sulphate) to a concentrated solution of ammonium nitrate and evaporating (305,760, Vol. XX, 258); a stable product obtained by evaporating (by spraying or passing over heated rollers) a liquor produced by addition of solid potassium nitrate to a concentrated solution of calcium, magnesium or ammonium nitrate, or urea (315,485, Vol. XXI, 155); and a non-deliquestent mixture of calcium and ammonium nitrates (316,664, Vol. XXI, 314).

Several inventions are directed to the production of fertilisers in granular form suitable for proper handling, storage and distribution. Such are, for example, a process comprising the use as a binding agent of a gelatinous phosphate of iron and/or aluminium derived from a rock phosphate (307,575, Vol. XX, 359), a process in which the fused material is allowed to fall in drops into a cold liquid (313,652, Vol. XXI, 57), a similar process comprising passing the molten material in thin streams or drops into a body of oil in which it is insoluble (314,639, Vol. XXI, 113), and a process of spraying a hot concentrated aqueous solution or a hot aqueous dispersion of the material into an ascending current of cold air (319,893, Vol. XXI, 479).

In connection with the subject of fertilisers may be further mentioned several inventions comprising the use of improved catalysts for ammonia synthesis, such as the use of a product prepared from double compounds of iron with cyanogen and alkalies, alkaline earths, etc., together with zirconium compounds (305,753, Vol. XX, 258), and the use (in the case of synthesis from nitrogen and steam) of ferrophosphorus obtained by heating a mixture of calcium phosphate, ferruginous bauxite, silica, and carbon (311,376, Vol. XXI, 34), or of such ferrophosphorus in presence of organic carbides or cyanogen compounds (314,894, Vol. XXI, 224).

### Vat Dyes

A vast amount of research on the part of the British, German, and Swiss manufacturers in respect of the vat dyes derived from anthraquinone is evidenced by the numerous specifications published in this field during the year. Benzanthrone derivatives in particular continue to receive close attention both as regards the production of new dyes and intermediates and as regards the improvement of the processes of production of the known dyes and intermediates of this series. Products derived from dibenzanthrone (violanthrone) and isodibenzanthrone (isoviolanthrone) have been especially numerous, and new methods have been described for obtaining dibenzanthrone (301,403, Vol. XX, 12; 303,538, Vol. XX, 129), halogendibenzanthrones (314,903, Vol. XXI, 224), isodibenzanthrone (303,454, Vol. XX, 129), and halogenisodibenzanthrones (315,276, Vol. XXI, 244). Much attention has also been given to dibenzpyrenequinone derivatives (307,926, Vol. XX, 507; 308,617, Vol. XX, 523; 310,891, Vol. XXI, 10), benzo- and naphtho-dianthrone derivatives (303,184, Vol. XX, 104; 307,364, Vol. XX, 359; 315,033, Vol. XXI, 135), anthranthrones (304,613, Vol. XX, 320; 305,489, Vol. XX, 360; 307,481, Vol. XX, 479; 311,347, Vol. XXI, 34; 316,149, Vol. XXI, 294), and anthraquinone-acridones (305,082,

Vol. XX, 233; 306,573, Vol. XX, 319; 311,283, Vol. XXI, 58; 314,899, Vol. XXI, 224). The dyestuffs include many condensation products, often highly complex or of unknown constitution, obtained from derivatives, especially amino-, halogen-, and carboxy-derivatives, of the benzanthrone and other polynuclear carbocyclic or heterocyclic parent materials.

#### Azo Dyes

Apart from the anthraquinone vat colours, the most fertile field for invention in the dyestuff industry is provided by the colours of the azo class. The azo dyestuffs suitable for production on the fibre (ice colours) continue to receive much attention. They are for the most part insoluble in water, and have as coupling components the well-known compounds of the Naphthol A.S. type which have a pronounced substantive character, viz., various arylides of 2:3-oxynaphthoic acid or of acetoacetic and like  $\beta$ -ketocarboxylic acids. The invention in the case of the monoazo and primary disazo products is usually, it appears, in the selection of the components, especially the diazo components, to secure special advantages, such as fastness to light, in the dyeings. New arylides of 2:3-oxynaphthoic acid, viz., the bis-(2'-oxy-3'-naphthoyl) arylene-1:4-diamines have also been described (311,194, Vol. XX, 570).

A few inventions relate to the production of stable diazo compounds for use in dyeing by the ice colour method; such are, for example, the fluorsulphonates (303,527, Vol. XX, 234) and borofluorides (316,691, Vol. XXI, 223; 317,355, Vol. XXI, 265).

#### Complex Metal Compounds of Dyes

A large group of inventions is concerned with the production in substance of the soluble complex metal compounds of azo dyes containing "chromable" groups, usually ortho-oxazo- or orthocarboxyazo groups. Chromium and copper are the metals usually introduced into the dyestuff molecule, but dyestuffs containing cobalt, nickel, manganese, iron, and some rarer metals, such as molybdenum, tungsten and uranium, have also been described. The products find their chief use as acid dyes for wool, but recent developments have included the production of dyestuffs of this class derived from components selected with a view to the direct dyeing of cotton, the dyeing of leather, the dyeing of regenerated cellulose silks, and the colouring of varnishes and lacquers.

Recent new intermediates for use as components for azo dyes include a number of complex condensation products containing heterocyclic rings, notably the cyanuric (1:3:5-triazine) ring (300,987, Vol. XX, 82), benzodiazines (310,076, Vol. XX, 506), thiazoles, quinoxalines, pyrimidines, etc. (305,487, Vol. XX, 360), and certain 2:3-benzocarbazole derivatives (317,928, Vol. XXI, 293), which couple with diazo compounds yielding azo dyes which on heating with dilute acids are converted into vat dyes of the  $\alpha$ -naphthocarbazole quinone series.

#### Dyes and Dyeing, General

Inventions relating to the use of new dyes or of specific known dyes for acetate silk have been less numerous than in some recent years. There are to be noted, however, besides the use of a few unsulphonated or monosulphonated azo dyes from selected components the use of such products as 4-amino-1:8-naphthalic acid imides substituted in the imide group (304,739, Vol. XX, 320), basic derivatives obtained from hydroxyanthraquinones (310,784, Vol. XX, 545), xanthone compounds (314,825, Vol. XXI, 135), *p*-amino-anilinoanthraquinones (315,905, Vol. XXI, 177), and *N*-anthraquinonylmorpholines (317,555, Vol. XXI, 266).

The discovery of new dyes, and the selection of known dyes, capable of yielding level shades on viscose silk, continues to be the object of a large group of inventions. The avidity with which this material takes up the usual substantive cotton dyes is a disadvantage from the point of view of the dyer, since it makes the range of colours which give dyeings free from uneven or streaky effects strictly limited. With few exceptions, the colours which are found to be suitable are of the azo class, especially such as are obtained by the use of 2:5:7-amino-naphtholsulphonic acid (*J*-acid) and *N*-substituted derivatives thereof as coupling components. A few complex metal compounds of azo dyes have also recently been found useful.

Several recent inventions relate to dyestuffs derived from 1:8-naphthalic acid. Thus acid wool dyes have been obtained by treating a sulphonic or sulphanic acid of 1:8-naphthalic anhydride or its derivatives with ammonia or an aliphatic or

aromatic amine (299,721, Vol. XX, 13; 309,552, Vol. XX, 594; 312,175, Vol. XXI, 90), and azo dyes have been obtained by diazotising similar unsulphonated 4-amino-1:8-naphthalic acid imide derivatives and coupling with arylides of 2:3-oxynaphthoic acid (302,173, Vol. XX, 159), while, as noted above, the unsulphonated derivatives are themselves suitable for dyeing acetate silk (304,739, Vol. XX, 320). 1:8-Naphthylene-naphthimidazole sulphonic acids for use as intermediates have also been described (316,143, Vol. XXI, 294).

#### Wetting, Dispersing, etc., Agents

The production of improved wetting, dispersing, emulsifying, and cleansing agents, usually sulphonic acids, for use in the dyeing and other textile industries, has received much attention. Such are, for example, sulphonated oxidation products of paraffin hydrocarbons, waxes, etc. (303,281, Vol. XX, 129), which are soap-like substances, sulphonated derivatives of halogenated unsaturated aliphatic or of unsaturated hydroaromatic hydrocarbons (303,917, Vol. XX, 158) which have high acid-resisting power, sulphonated products from alcohols containing more than eight carbon atoms, e.g., cetyl alcohol (307,709, Vol. XX, 479; 317,039, Vol. XXI, 238), and products obtained by sulphonating fatty acids in presence of salts of acetic acid or its homologues (312,283, Vol. XXI, 338).

#### Acetic Acid and Anhydride

The present industrial importance of these materials is reflected in the large number of inventions relating to their production and purification. Processes of production are based mainly on the oxidation of acetaldehyde by air or oxygen, in presence of a catalyst and usually in acetic acid solution (304,350, Vol. XX, 188; 308,937, Vol. XX, 431; 319,433, Vol. XXI, 387), on heating acetylene with catalysts in presence of oxygen and hydrogen, with precautions against explosion (304,855, Vol. XX, 213), on extraction from pyroigneous acid (316,284, Vol. XXI, 295), and on reaction of dimethyl ether with carbon monoxide in presence of sodium methylate or ethylate or alkali formates (319,030, Vol. XXI, 361).

Numerous methods of extracting pure acetic acid from its aqueous solutions have been described, and the following are examples of the methods used:—Extraction with or rectification in presence of various organic solvents such as trichloroethylene (300,923, Vol. XX, 82), amyl acetate (301,415, Vol. XX, 130), methylene chloride (301,734, Vol. XX, 130), a mixture of quinoline, dimethylaniline, and certain hydrocarbons (303,494-5, Vol. XX, 234), chloroform, ethyl chloride, or methyl chloride (315,397, Vol. XXI, 244); extraction with a solvent such as amyl acetate and distillation of the extract with a liquid, e.g., petroleum, forming with the acetic acid a binary mixture of minimum boiling point (317,462, Vol. XXI, 362); and extraction with various solvents at temperatures below  $-20^{\circ}\text{C}$ . (308,210, Vol. XX, 507).

As methods of preparing the anhydride may be mentioned the dissociation of the acid vapour by heat in presence of a catalyst (301,562, Vol. XX, 30; 316,898, Vol. XXI, 243; 318,662-4, Vol. XXI, 337), fractional condensation from acetic acid vapour by the use of benzene, carbon tetrachloride, petrol, etc., to carry away the water vapour (303,772, Vol. XX, 158), heating the acid in presence of a stream of hydrogen in vessels lined with certain metals, and separating the anhydride from the vapours by fractional condensation or by the use of suitable solvents, entraining vapours, or water-binding substances (305,147, Vol. XX, 233), and heating the acetic acid vapour in presence of sulphur dioxide (313,418, Vol. XXI, 57).

#### Foreign Bankruptcy Laws

THE Overseas Committee of the F.B.I. have set up a sub-committee to examine the position of the Bankruptcy laws in foreign countries, with a view to seeing whether there is no possibility of effecting improvements. In many countries the laws relating to bankruptcy and suspension of payment are highly unsatisfactory to British and other traders, and the Federation feel that if the matter is properly examined they may be able to make some proposals for amending the position. The institution of this sub-committee coincides with the report from the F.B.I. representative at Havana, calling attention to the abuse and consequent loss incurred by British firms on account of the present laws in Cuba affecting debt collection and suspension of payment.

## The Association of British Chemical Manufacturers

### A Survey of Its Activities and Those of Related Bodies

THE Association of British Chemical Manufacturers has had another very active year under the chairmanship of Dr. G. C. Clayton (of Imperial Chemical Industries, Ltd.), who has been re-elected for a further year. Dr. E. F. Armstrong has succeeded Lord Melchett as vice-chairman, while Mr. E. V. Evans, of the South Metropolitan Gas Co., has accepted the post of honorary treasurer vacated by Dr. E. F. Armstrong. Sir David Milne-Watson, of the Gas Light and Coke Co., and Mr. C. A. Hill, of British Drug Houses, Ltd., have been re-elected president and vice-president respectively. The membership of the Association remains practically unchanged, a few resignations having been made good by the accession of new firms.

#### Legislation and Tariffs

No serious problems have arisen in regard to legislation during the year, but there have been a number of difficulties in connection with tariffs. The blasts of trumpets at Geneva have not caused the tariff walls to fall or even to tremble; in fact, increases have occurred in various directions which have had an adverse effect on British trade. The latest proposals for a tariff holiday may have more success, though on the other hand we have the example of America, which has been able to develop her industry in a surprising manner owing to the security offered by her flexible tariff system. The fact that America is becoming a serious competitor all over the world and is steadily obtaining a growing share in the foreign trade is likely to induce countries that desire to protect their home industries to increase the tariff barriers still further.

As a practically free trade country, we are badly situated when we have to compete against nations who, with their internal markets secure, can dump their surplus production abroad at prices against which even our Safeguarding of Industries Act, where applicable, fails to provide adequate protection. This is becoming a serious menace, and the Association welcomes any proposal which will help in developing our export trade and in protecting us against dumping. Even inside the Empire the tariff position has given cause for anxiety. The subject is very complicated, as was seen when the change in the basis of the Canadian Preferential Tariff took place, by which the percentage of the value of the material and labour in a product which must be British has been raised from 25 per cent. to 50 per cent. Though the underlying idea is highly commendable in the interests of Empire trade, the change has resulted in a number of cases in which British manufacturers are penalised owing to the fact that the raw materials themselves are unobtainable commercially in the Empire, and constitute 50 per cent. or more of the value of the final product. Strong representations have been made to the Canadian authorities, and these are still under investigation.

#### Safety in the Chemical Industry

It was mentioned last year that the Association was engaged in drawing up regulations to serve as a model on which to frame safety measures in the Chemical and Allied Industries. Part I, entitled "Model Rules," was issued in provisional form in the early part of the year and had a very good reception. This consists of the safety rules in concise form, and enunciates the general principles which have to be observed in planning safeguards in the industry with a view to reducing the risks of damage to personnel and material. The preparation of Part II is now actively in hand; this will consist of a more extended explanation of the objects to be attained by the rules, the dangers arising from their non-observance, and the best or most convenient methods of obtaining the desired results.

The underlying idea is to bring together in a convenient form such data as are necessary to enable the manufacturer to give effect to the principles set forth in the model rules. This work is being done by a strong representative committee of technical experts in collaboration with the Home Office departments concerned with safety in industry. All likely sources of information are being explored in this country, and an examination is being made of the safety regulations in operation in the main chemical manufacturing countries of the world.

As part of the safety problem the Association has initiated

a service of safety circulars. These are based primarily on accidents which have occurred in chemical and similar works, and contain a short account of the accident and its causes, followed by concrete suggestions as to the precautions which will obviate similar occurrences in future. Special attention is given to accidents from which useful lessons can be drawn or which arise from unusual or unexpected causes. There has been a big demand for these circulars, thus showing that they are supplying a definite want, and the distribution has risen to 1,500 copies. Further schemes are under consideration for the extension of this work on a national and international basis.

#### Propaganda

The Chemical Section of the 1929 British Industries Fair, which was organised by the Association, proved as attractive as in former years, in spite of the intense cold, which was responsible for keeping away many potential buyers, especially from abroad. As an experiment, the Association decided to arrange for organised visits of chemical teachers and senior students to the Chemical Section, where they were given short discourses by the various exhibitors regarding products and processes likely to be of educational value. The Association felt that by taking this course it would be stimulating and maintaining the connection between research and industry, and that the younger generation of students who will become the teachers and research workers of the future would have a unique opportunity of learning the latest achievements of British chemistry and of appreciating that the British chemical industry is now second to none.

The response to the Association's invitation was so gratifying and the visits so much appreciated, not only by the visitors but also by the exhibitors, that it has been decided to make these arrangements a regular feature of future fairs. Plans are now well advanced for the fair at Olympia in February, 1930, and the Chemical Section will be as interesting as ever, so that any members of the teaching profession who wish to arrange a conducted tour and did not participate in the 1929 programme are advised to make early application to the Association.

The Association has made a study of Continental fairs, and it was gratifying to find that the Chemical Section of the British Industries Fair was far ahead of any other as regards the number, variety, and technical interest of its exhibits.

Propaganda has also been conducted at the Barcelona Exhibition, where arrangements were made for the distribution of the Association's new directory. This directory has several improved features, and is invaluable to anyone who wishes to buy British chemicals, as all the names listed are those of actual makers. As it is in six languages, it is admirably suited for propaganda in foreign countries, and a wide distribution has been made abroad, and especially throughout the Empire. A new directory of British Fine Chemicals, showing the manufacturers of some 3,000 fine chemicals, is now ready, and will be issued widely throughout the world.

#### Patents

The Patents Committee of the Association has been very active since the British Science Guild report on Patent Law Reform was published at the end of last year. When the appointment of a Board of Trade Committee, to investigate the whole patent position, was announced in the early summer, the Association arranged, through the Federal Council for Chemistry, that the other chemical bodies who were interested should collaborate with them in drawing up a memorandum to represent the views of chemistry. This memorandum has received the endorsement of the councils of the main chemical institutions, who thus speak with one voice on the subject, and has been presented to the Board of Trade Committee, before whom members of the Patents Committee have given evidence in support of the proposals.

The memorandum, which will shortly be published, deals primarily with those points that are of greatest importance to chemical science, and the proposals are aimed at correcting various anomalies and abuses of our patent system, which were never intended by the legislators, in order to improve the position of the holder of a British patent. The section that

will probably be of the greatest interest is the one dealing with patents for inventions subserving medical purposes, where a scheme has been submitted which promises to reconcile the objection of the medical profession to patents for medical inventions on ethical grounds, with the need for adequate safeguards for the industry in developing new methods of treatment to the ultimate advantage of the public.

#### Miscellaneous

Only a passing reference to some of the other activities of the Association is possible. Schemes for the collection of production statistics in various branches of the industry are well in hand. The issue of a monthly summary of information on chemical trade has been commenced as a regular feature, with the intention of bringing together, in a readily accessible and convenient form, the information published in a variety of journals bearing on chemical trade, such as import and export statistics, tariffs, production data, etc. The interests of members have been closely watched in transport questions, and various concessions have been obtained in regard to rates and regulations for rail traffic which should be of considerable financial benefit to members. The Association also takes an active part in the work of other organisations such as the Federation of British Industries and the International Chamber of Commerce, and is in relationship with the corresponding organisations in the main chemical producing countries.

To conclude, the year's activities have shown conclusively how useful a trade association can be to its members, and how necessary it is to maintain such a body for watching the general interests of the industry.

### British Chemical Plant Manufacturers

The British Chemical Plant Manufacturers' Association has had a very successful year, and its membership is now considerably higher than it has ever been. Mr. J. Arthur Reavell, of The Kestner Evaporator and Engineering Co., Ltd., consented, at the pressing request of his colleagues, to serve as chairman for a third year. Dr. H. Bush, of Huntingdon Heberlein and Co., Ltd., has become vice-chairman, while Mr. E. A. Alliott, of Manlove Alliott and Co., Ltd., continues as honorary treasurer.

The publicity and propaganda work of the Association on behalf of British chemical plant has continued on a steadily increasing scale at home and abroad, and steps are now being taken to develop the Empire markets. In this work the Association has made good use of its new directory, produced in the early part of the year, and classified in such a manner as to constitute a valuable buyers' guide to anyone requiring plant for the chemical or allied industries. Nearly 3,000 copies have already been distributed and *bona fide* applicants can obtain a copy gratis from the Association's office at 166, Piccadilly, London, W.1.

The Association also participated in the British Industries Fair, and had an office in conjunction with the Association of British Chemical Manufacturers, from which its directory and the trade literature of its members were supplied to enquirers. This arrangement will again be adopted at the Fair at Olympia in February next year.

### Chemical Engineering Abstracts

A monthly summary of information on chemical engineering has recently been commenced with the object of collecting, in a convenient form, abstracts of the more important papers and publications bearing on the subject of chemical plant. This service, which is the first of its kind, appears to supply a very definite want, and has received such warm appreciation from the members that steps have been taken to put the idea on a permanent and wider basis by co-operating with the other bodies interested in abstracts relating to chemical engineering.

The work of the Resistant Metals Committee is still continuing and important developments may be anticipated in this direction in the near future. The Association is in direct touch with related activities in this country and with the corresponding organisation in Germany, so that it is in a position to watch all new developments there.

With its increased membership, the Association promises to perform even more useful work in the future than it has done in the past.

### Association of Tar Distillers

The Association of Tar Distillers, which works in conjunction with Group V of the Association of British Chemical Manufacturers, has had another useful year. Mr. W. J. U. Woolcock, who had been honorary secretary for the past ten years, resigned the position in the early part of the year, and Mr. J. Davidson Pratt, the general manager of the Association of British Chemical Manufacturers, was appointed in his place.

The statistical scheme, of which mention was made last year, has received increased support, and the Association now has at its disposal data in regard to the production of the various primary products of coal tar.

Special attention has been given during the past few months to the development of new uses and outlets for creosote in view of the largely increased production of this oil. A Creosote Propaganda Committee has been formed and has been active in exploring all possible means of opening up new markets, both at home and abroad. The most important outlet at present is for preserving timber, and since this country has to import the bulk of its wood supplies, there is great need for a campaign to educate users of wood and the public generally to the necessity for the preservation of timber employed for constructional and other work. Similar propaganda is required in foreign countries, where there is a big wastage of wood from rapid deterioration, due to its being used in an untreated state.

The important work of standardising tests for tar products as applied in regular works practice and in the buying and selling of tar fractions, which was initiated by the Joint Fuel Committee, and with which the Association has been closely connected, has been completed, and the report entitled "Standard Methods for Testing Tar and Its Products" is now on sale. This represents a valuable piece of work, and the report should be in the hands of everyone who deals with tar products. In view of the difficulties involved in standardisation work of this nature, the greatest credit is due to all who have contributed to the production of the report. They have set an example which might be followed with great advantage by other sections of the chemical trade and by other branches of industry.

Useful work with which the Association has been concerned has also been done by the British Engineering Standards Association in producing standard specifications for road tar, and by the National Benzole Association in respect of specifications for benzole, toluole, xylol, and naphthas. The latter were published at the same time as the standard tests mentioned above, with which they are intimately connected.

### British Disinfectant Manufacturers

The old British Disinfectant Manufacturers' Association was dissolved in May of this year, but recent events in connection with Government specifications for disinfectants have indicated the necessity for some organisation to watch the interests of the disinfectant manufacturing industry. Steps have, therefore, been taken to form another Association on a new basis, and there is every hope that by the time this notice appears, an organisation representative of the whole of the industry will be in existence.

### Ammonium Phosphate as Fertiliser

"PHOSPHATES TUNISIENS," which was the first concern to begin the production of synthetic nitrate of lime in France, has just perfected the production of fertiliser grade phosphate of ammonia. Phosphate from the company's mines in Kalaa-Djerda, Tunisia, is shipped through the port of Bayonne to the new Pierrefitte-Nestalas plant (in the Hautes-Pyrénées) and is transformed into phosphoric acid by an electric furnace process. The acid is saturated with ammonia from the Soulom plant, yielding ammonium phosphate. A few necessary mechanical improvements will be effected soon, and toward the end of the year, it is expected that the plant will operate at full capacity of from 15,000 to 20,000 metric tons annually. The quality of the product obtained is said to be satisfactory both from a chemical and physical point of view. The "Phosphates Tunisiens" is now considered as ranking among the most important French fertiliser and chemical companies. —(U.S.A. Assistant Commercial Attaché, D. J. Reagan, Paris.)

## Chemical and Allied Societies: Their work during the Year

*The following specially contributed notes on the work of the principal chemical and allied societies indicate both the range and the importance of their work, and record many interesting developments during the year.*

### British Engineering Standards Association

THE British Engineering Standards Association, which was founded in 1901, and received a Royal Charter during the past year, is the national standardising body for the technical industries of the country.

The Association has recently been reorganised as a result of having obtained the Royal Charter. The main features of the new organisation are that the Council will now have a majority of elected members, and that the work is divided into twelve or fourteen Industry Sections, each under the control of an Industry Committee. These Industry Committees are very fully representative, and are responsible under the Council for the whole of the technical work of their particular branch. Before them come all requests for standardisation, and they have to ascertain by calling a Conference or otherwise, that the work proposed is to fulfil a recognised want, and whether the necessary funds are available for carrying out the work. The Association employs a specialised staff who act as a liaison between all the Committees, and carry out the ideas of the Technical Committees. These Committees are also assisted in many cases by the technical officers of the firms up and down the country, and it has been estimated that the indirect support of industry to this work is somewhere in the nature of between twenty and thirty thousand pounds a year. It must be remembered also that between two and three thousand members throughout the country attend the meetings, and give their time and experience without fee or recompense.

Among the work at present nearing completion may be cited the British Standard Specification for the Grading of Sizes and Methods of Use of Sieves and Screens. This work was undertaken at the request of the South African branch of the Association, the War Office and the Heat Engine and Boiler Trials Committee of the Institution of Civil Engineers.

The British Standard Specification for Gas Cylinders, based on the Home Office recommendations of 1895, is under consideration. This work was undertaken at the unanimous request of a conference of manufacturers and users. The specification will be limited to those technical clauses necessary for the purchase of gas cylinders, and the Committee will have the assistance and experience of those responsible for the Home Office Report, and also the members of the Gas Cylinder Research Committee of the Department of Scientific and Industrial Research.

There is quite a large number of British Standard Specifications issued by the Association, which are of interest to the chemical industry, and which are included in the Association's published lists.

### The British Association of Chemists

THE association has made very satisfactory progress during the past year. This is reflected in the increase in membership (109), the largest recorded for many years. The Unemployment Benefit Fund is in an even stronger position than last year, in spite of the heavy calls made upon it. The sum of £504 has been paid out in benefits during the current year. The increase is due partly to the increase in membership and the consequent number of units taken up, and partly to the temporary unemployment produced by amalgamations in industry. Not the least important aspect of this form of insurance is the effect upon professional salaries and status. In a large number of cases it has enabled chemists to refuse an inadequate salary and thus assist in raising the economic status of the chemist.

The appointments bureau has extended its activities and the number of employers who have approached the bureau direct has increased by 300 per cent. Working in co-operation with the Unemployment Benefit Fund, it has been able to negotiate very satisfactory conditions in regard to salary. A number of appointments, carrying salaries ranging from £700 to £1,000 per annum and over, have been secured for members. The Legal Aid Department examines an increasing number of agreements every year, and continues to give legal advice to members, particularly in case of insufficient notice.

A large number of cases have been submitted, all of which have been settled on the basis of three months' notice or more, or equivalent compensation, agreeably to the legal precedent established by the Association, without recourse to litigation.

The association has been continually in touch with the Ministry of Labour in all cases relating to the employment of foreign chemists in Great Britain. The association has thus been able to render assistance to the Ministry and to British chemists in putting into practice the Aliens' Order. The Council is completely satisfied that in no case has a foreign chemist been employed where a suitable appointment of a British subject could have been made. The association's recommendations in regard to the organisation of the profession are still receiving the close attention of the Council and the Registration Committee. The preliminary £100 called for, as a nucleus of a registration fund, was quickly subscribed or guaranteed, an indication of the interest taken by members in the whole question. It remains to ascertain the views of the other societies and to consider further action along the lines suggested in the annual report.

The Council has realised that ultimately the recognition of the profession as a legal entity depends upon the opinion of the public, so that the Council has initiated and encouraged publicity in every possible way. The association has succeeded in obtaining considerable publicity for the profession in the public press all over the world. It has not infrequently been of a kind which, from a technical point of view, leaves something to be desired, but it is nevertheless true that while the standard of scientific education of the public remains rudimentary a more elaborated method of publicity is impracticable. The most that can be expected, and this the association claims to have accomplished, is the awakening of the public interest in chemistry, thus encouraging it to turn for more detailed information to those best qualified to instruct it.

The *Chemical Practitioner*, the official organ of the association, has been published regularly during the past year. It has kept members in close touch with the society's activities and has proved valuable as an instrument of publicity.

### The Chemical Industry Club

FOUNDED in 1916 to afford a social centre where those engaged both in research and experimental work and the application of the results of such investigations to industrial and commercial use, could meet on a democratic basis for interchange of information and ideas, the Chemical Industry Club is in its way unique among those associations and bodies in this country, the interests of which are scientific or scientific and commercial.

Commencing with occasional informal meetings held in private chambers and in London cafés, its originators—a small body of enthusiastic professional and business men—pushed their ideal of such a centre to its logical conclusion when, in 1918, premises were obtained at 2, Whitehall Court, adequate for the formation of a comfortable club with all the amenities of club life. It appears to have been the concern of the various chairmen and committees who have guided the policy of the club to its present success that whilst the membership has for some time stood in the neighbourhood of 700, the average number of those who enjoyed the advantages offered by the Club was far below the capacity of its premises. It has been suggested that the chemical industrial world was not generally a "social" one. For ourselves we would rather suggest that it is one to which the amenities of club life had hitherto been unknown. Not the least of these amenities may be reckoned that of a pleasant unsociability, and it is possible that the artificial brightness of the "chatty" "Conversation" or "Reception" or the "question time" after a lecture possesses a greater appeal to those for whom a defined programme and a prearranged date are the indispensable incentives for the desertion of the domestic hearth, than the more occasional atmosphere of club gossip and saddlebag arm-chairs. Experimentally, the committee therefore in-

augured a series of lectures and lantern slides, and by the adventitious aid of such innocuous amusements through increasing intervals, have stimulated their members to a growing appreciation of club community. It is probable that few of our readers have not visited the Club, but for that few we would explain that the premises comprise smoking room, billiard room, card room and dining room. All of these have recently been redecorated and largely refurnished. The Club possesses a number of really admirable modern etchings presented by different members.

The Club has become in the chemical industrial world the recognised meeting-place where, over a smoking-room talk, the politics of the industry and its allied associations and societies may in an atmosphere of equality be discussed and unofficially formulated. We are by no means certain that this opportunity of democratic fireside discussion which the smoking room of the Chemical Industry Club affords, may not already be one of the most important factors for the future of the chemical industrial world.

"For sometimes in a smoking room through clouds of 'ers' and 'ums'

Obliquely and by inference illumination comes,"  
and the needs or grievances of one group are comprehended by the other.

### The Faraday Society

THE society had an unusually successful year from the point of view of the papers which were communicated. The number of pages of the Transactions was greater than at any previous time, the number of papers being 102 compared with 78 last year. There were held during the year three of the general discussions for which the society is so famed; the first inaugurated by Professor V. M. Goldschmidt dealt with Crystal Structure and Chemical Constitution, the second followed the reading of Dr. J. C. Hudson's Third Experimental Report to the Atmospheric Corrosion Research Committee (British Non-Ferrous Metals Research Association), and the third was devoted to "Molecular Spectra and Molecular Structure." The last mentioned meeting was held in the University of Bristol, where the Society had the pleasure of being the first occupants of the delightful hostel which is now occupied by the fortunate students of that University; visitors came from all parts of the world—not excluding America, India and Japan.

The Society, largely by reason of its successful general discussions, has always had strong affinities with societies dealing with kindred subjects on the continent and in America; for more than twenty years reciprocal arrangements for the supply of transactions on "most favoured" terms have existed between the society and the American Electrochemical Society. At the time of the Jubilee of the society in November, 1928, conversations took place between representatives of the two societies and the Deutsche Bunsengesellschaft with a view to increasing the international facilities, as a result of which definite arrangements have now been concluded whereby a member of any of the three societies may acquire membership of one or both of the others without paying an entrance fee and with a substantial reduction of the corresponding annual subscription. The scheme was only brought into action late in the present year, but useful results have already followed.

### The Chemical Society

DURING 1929 the Chemical Society has continued to pursue the objects for which it was formed in 1841, namely, "The general advancement of chemical science by the discussion and publication of new discoveries, and the interchange of valuable information regarding them." Thirteen ordinary scientific meetings have been held at which 53 papers were read and discussed, and up to the end of November the *Journal of the Chemical Society* (published on the last day of each month) contains 355 papers occupying 2,661 pages. Abstracts "A" (Pure Chemistry) for 1929 contained up to the end of November 1,348 pages as against 1,292 at the corresponding period in 1928. The Annual Reports for 1928 (published in February 1929) included reports on Sub-Atomic Phenomena and Catalysis, as well as the usual reports.

In addition to the ordinary scientific meetings, the Society has held three special lectures. On April 25, Sir Harold B. Hartley delivered the Theodore W. Richards Memorial Lecture. The first lecture in honour of the late Sir Alexander Pedler (a generous benefactor to the Society) was delivered by the late Professor W. H. Perkin, on May 30, and on December 12 Professor Dr. H. Freundlich gave the second Liversidge Lecture and chose as his subject "Surface Forces and Chemical Equilibria."

Following the practice of the Council to hold from time to time its anniversary meetings in the provinces, the annual general meeting was held in the University of Leeds on March 21; the anniversary dinner taking place the same evening in the Town Hall, Leeds. The company present, which numbered 282, included Lord Lascelles, and many other distinguished guests.

Grants for research work amounting to £736 have been awarded by the Council during the year. The advantages of the Library, which contains some 33,500 volumes, and is open from 10 a.m. until 9 p.m. every week-day except Saturday, when it closes at 5 o'clock, are available to Fellows of the Society and to the members of the following institutions which make contributions towards its maintenance: The Association of British Chemical Manufacturers, the Biochemical Society, the Faraday Society, the Institute of Brewing, the Institute of Chemistry, the Society of Chemical Industry, the Society of Dyers and Colourists, the Society of Public Analysts. During 1928, there were 8,037 attendances, of which 2,669 were made by members of the contributing institutions; the number of books borrowed was 5,262, and the additions to the Library numbered 945 volumes. Conditions of Fellowship can be obtained from the Assistant Secretary, The Chemical Society, Burlington House, Piccadilly, London.

### The Fertiliser Manufacturers' Association

THE Fertiliser Manufacturers' Association, Ltd., of Great Britain, is, as is no doubt generally known, one of the oldest trade organisations in this country, and there can be no doubt whatever that it is the oldest association of fertiliser manufacturers in the world. Founded in 1876 under the title of The Chemical Manure Manufacturers' Association, the name was changed in 1904 to The Fertiliser Manufacturers' Association, and in 1919 it became a company limited by guarantee of its members in order that it might take over from the Government the task of providing superphosphate makers with phosphate rock during the difficult post-war period. Naturally the primary function of the association is to act as the central negotiating unit on behalf of the industry in regard to all questions which affect it as a whole, such, for example, as legislation, railway carriage conditions and rates, harbour and dock dues, and kindred matters.

The past year has been particularly important in regard to legislation affecting the industry, in proof of which it is only necessary to mention two Acts of Parliament, viz.: Rating and Valuation (Apportionment), and Agricultural Credits. The former is certainly one of the most far-reaching enactments of modern times and the benefits accruing from its coming into operation should have a marked influence on the competitive power of British Industry in the future. The Agricultural Credits Act, too, is of great importance, and it is an open secret that this has been very much more widely used by farmers than was anticipated.

The association amongst other activities also discharges the function of providing the official statistics of superphosphate production and consumption for the country as a whole. It may be of interest to observe that the total consumption of superphosphate in Great Britain and Ireland during the fertiliser year ended June 30, 1929, was 724,178 tons, an increase of 53,214 tons on the preceding year. Production, too, showed an equally marked advance, the tonnage made in British and Irish factories during the same period being 556,370 tons or 59,809 tons more than the previous twelve months. The market showed some extraordinary variations, prices on the continent starting from a very low level and finishing at the highest reached for many years past. In Great Britain prices, though rising slightly, remained relatively stable; they neither fell so far at the commencement nor rose so much at the end of the year. It remains to add that the

outlook is not very promising for the forthcoming fertiliser consuming season owing to the very low prices farmers are now obtaining for their grain and potatoes.

### The Institute of Brewing

COMMENCED IN 1920, the Institute of Brewing Research Scheme is financed entirely by the annual subscriptions of corporate members of the Institute. The fund so formed is administered by the Research Fund Committee for the promotion and assistance of research for the benefit of the fermentation industries generally. The following subjects have received attention:—Timber for casks; the effect of hydrogenation concentration in the various stages of the brewing process and on the nutrition of yeast; agricultural and other aspects of barley and hops; and the chemical and biological investigation of the preservative substances in hops (first under Professor F. L. Pyman and later under Dr. T. K. Walker). The Research Committee has also appointed a biochemist to investigate the proteins of barley and malt and the changes in them which occur during malting and brewing. This work is being carried on at Rothamsted. The carbohydrate constituents of barley and malt are being dealt with by Professor A. R. Ling, at the British School of Malting and Brewery, Birmingham.

### The Institute of Chemistry

It may be anticipated that the annual report of the Council of the Institute will show a further substantial increase in the roll of Fellows and Associates and Registered Students and a good record of useful work. The Council has been in communication with the professors and heads of chemical departments of universities and colleges, suggesting that students intending to adopt chemistry as a career should have regard not only to their science but to the professional aspects of their subject. In pursuance of this suggestion, lectures will be delivered from time to time on the practical life of the chemist and the prospects of his profession in its various branches. These are being given by professors and well-known practitioners, and, in some institutions, by the permanent officials of the Institute, who are also co-operating with the Ministry of Labour in meetings held at schools, where boys on the science side are addressed on science as a career. Among other matters which have come under the particular attention of the Council may be mentioned the interests of chemists in the beet sugar industry, the relations of private practitioners and research associations, and the regulations regarding the distribution of methylated spirits.

Early in the year, the Lord President of the Privy Council notified the Institute that a petition for the grant of a Royal Charter had been presented to His Majesty in Council by the Australian Chemical Institute. Believing that the chief aims and objects of the Australian Institute to be practically identical with those of the Home Institute, and, as such, entirely laudable and deserving of recognition, the Council of the Home Institute, on the report of its legal and parliamentary committee, felt disinclined to oppose the petition, but expressed the view that where Royal Charters were granted to several bodies concerned with the qualifications for the practice of any profession within the Empire, the standard of the requirements for membership should be approximately the same. The Home Institute, therefore, expressed the earnest hope that in the event of His Majesty granting the petition of the Australian Chemical Institute, adequate safeguards would be provided to ensure that the standard of qualification for its Associateship and Fellowship, respectively, should be comparable with that required by the Home Institute. The legal and parliamentary committee has also dealt with the reform of the British patent system, the Local Government (Scotland) Bill, in addition to matters such as the rights of inventors and chemists' contracts of service, which are still under consideration.

The local sections of the Institute, both at home and in the Overseas Dominions, have been active, and many papers of interest have been read at their meetings. A new section has been formed for Aberdeen and the North of Scotland. The statistics of the Appointments Register tend to show that, on the whole, the output of chemists from the universities and

colleges is being steadily absorbed. The number of unemployed chemists has been proportionately rather less than it was during 1928.

The benevolent fund committee is again able to record an increase in the number of annual subscribers, and in the total amount of the contributions received during 1929 compared with those received in 1928, but there has been a considerably greater increase in the amount of the annuities and grants disbursed.

The Meldola Medal for 1928 was awarded to J. A. V. Butler, D.Sc.(Birm.); the Sir Edward Frankland Medal and Prize for registered students were won by Cyril Fryer, of Birmingham; the Pedler Scholarship, awarded to Mr. George Morrison Moir, is being extended until June, 1930. The Streatfeild Memorial Lecture was delivered in November by Mr. Lewis Eynon, his subject being "The World's Sugar Industry." The first S. M. Gluckstein Memorial Lecture was given in December by Dr. L. H. Lampitt, who dealt with "The Chemist and Commerce."

### The Institute of Metals

AN outstanding event in the history of the Institute of Metals took place in March last when the Institute celebrated its coming-of-age. Founded in 1908 with an initial membership of only 250, by March, 1929, the membership totalled 2,050. The celebrations took the form of a banquet at the Trocadero Restaurant and a conversazione at the Science Museum, South Kensington, both of these functions being largely attended. In May the Annual May Lecture was given by Sir Oliver Lodge, on "States of Mind which make and miss discoveries; with some Ideas about Metals."

Another very notable event in the history of the Institute was the holding—in September—of the first meeting of the Institute in Germany. This gathering was extraordinarily successful, attracting several hundreds of members from no fewer than 18 different countries. Papers of an exceptionally important character were presented, and visits were paid to works in the neighbourhood of Düsseldorf and Berlin, whilst a whole-day trip to Essen was an outstanding feature of the visit.

The work of the six local sections of the Institute progressed very favourably, and a full programme has been arranged for the winter session.

During the year the Institute has issued two 800-page volumes of Proceedings, in which are incorporated all the papers presented at the March, May and September meetings, together with an invaluable series of abstracts—numbering many thousands—prepared from metallurgical publications published throughout the whole world.

### The International Superphosphate Manufacturers' Association

It is only three years since the International Superphosphate Manufacturers' Association was brought into being, yet in that relatively brief period it has attracted to its membership twenty-nine countries, representing rather more than two-thirds of the world's superphosphate consumption, the total of which in round figures was not less than 14½ million tons for 1928. It follows, therefore, that, the Association is representative of a production and consumption of superphosphate attaining to about ten million tons annually.

The chief task of the association and its constant pre-occupation is propaganda, for it is very well known that great as is the world's consumption to-day, it can be very largely increased. If all the countries represented by the association could bring their consumption of superphosphate per acre up to the level of the highest, the maximum manufacturing capacity now in existence could not meet the demand. Propaganda as conducted by the association takes several forms, one of the most useful of which is the fact that it provides a means of exchanging ideas. Thus, for example, if France produces a striking series of posters, specimens are at the disposal of each member country; if the German industry evolves a new form of appealing to the farmer, information as to the method adopted becomes available for all. The most important of the association's propaganda activities is the Hamburg Experimental Station, where under the able direc-

tion of Dr. C. Krügel, experimental work on strictly scientific lines is conducted. It would be impossible adequately to describe in the space at our disposal the work of the station, and it must suffice to say that both pot and field cultural experiments in all crops are constantly carried on in order to determine the best fertiliser combinations for the most important of the farmers' crops under various climatic conditions, several of which can be duplicated by the means at the disposal of the station.

Mr. E. G. Martens, the president since the foundation, is a well-known figure in fertiliser circles throughout the world, and under his leadership the member countries are rapidly getting to appreciate each others' problems and good fellowship is taking the place of former misunderstandings. The honorary secretary is Mr. A. N. Gray. The association publishes a periodical each month entitled "Superphosphate," which has a world-wide circulation. It is printed in three languages, English, French, and German, and besides recording the work of the Hamburg station, it gives information in connection with the results of using superphosphate not only from all parts of Europe but from America, Africa, Asia and Australia.

The International Superphosphate Manufacturers' Association constitutes a veritable League of Nations to increase the use of the product its members manufacture; it forms an international parliament of the industry; it pools and exchanges ideas, and finally (and perhaps that is not its least important function), it collects and disseminates among its members reliable statistics as to production and consumption. It seeks ultimately to include amongst its members every manufacturer of superphosphate throughout the world, a very laudable ambition which is in a fair way to be realised.

#### The Institution of Chemical Engineers

DURING 1929 the progress of the Institution of Chemical Engineers is reflected in an increase of membership of about 14 per cent. At the annual corporate meeting held in March, the retiring president, Sir Alexander Gibb, gave an address on "The Co-ordination of Engineering Institutions and Societies," a subject to which he has devoted much attention. The address, which evoked widespread comment, drew attention to efforts made, both in this country and elsewhere, to attain such co-ordination. Mr. J. Arthur Reavell was elected president for 1929, with vice-presidents Mr. C. S. Garland and Mr. F. Heron Rogers. Professor J. W. Hinchley and Mr. F. A. Greene were elected hon. secretary and hon. treasurer respectively.

Public lectures delivered during the year included one in January by Professor John W. Cobb, of the University of Leeds, on "The Reactivities of Solid Carbon in Fuel Processes," and one in October by Dr. W. H. Hatfield, of Sheffield, on "The Fabrication of Acid Resisting Steel Plant." In connection with the latter, Dr. Hatfield showed excellent cinematograph films which had been made specially for the occasion. A two-day conference on "Vapour Absorption and Adsorption," held in December, drew large attendances

at all sessions, and evoked lively discussions. Other papers during the year included one at the annual corporate meeting, when Professor B. P. Haigh spoke on "Chemical Action in Relation to Fatigue in Metals," and one in November, by Mr. James Strachan, on the "Production of Cellulose in the Paper Industry." Prior to the reading of the latter paper, a party of members had the privilege of visiting the British Vegetable Parchment Mills at Northfleet, through the kindness of Mr. William Harrison and his co-directors.

The President's reception, held on November 6, again attracted a large and distinguished gathering, over 400 members and friends being received by Mr. and Mrs. Reavell. The Graduates and Students' Section held four meetings, at one of which an address was delivered by the President on "Some Aspects of Chemical Engineering." The Associate-Membership examination of the year was noteworthy in that the "Home" paper was worked in February and March, thus allowing a longer interval until the written examination in London, held in July. As usual, the *viva voce* session proved of considerable value to the examiners, and, it is felt, to the candidates. Towards the end of the year, recognition was granted to the courses in chemical engineering at King's College, London, so that students passing creditably through this course would be exempted from the Associate-Membership examination of the Institution. Eight papers were presented to the World Engineering Congress at Tokio, at which Sir Alexander Gibb was the chief representative of the Institution.

During the year, awards have been established for services rendered to the Institution. The Moulton Medal in gold, commemorating the association during the war of the late Lord Moulton with chemical engineering, will be awarded for the best paper of a mature character read before the Institution and published in the Transactions. A junior award, in silver, with a prize of books, will be given to the best paper of the year communicated by a graduate or a student of the Institution. A further medal, with which the name of the late Professor Osborne Reynolds will be associated, the gift of Mr. F. A. Greene, will be awarded annually for the most meritorious contribution to the progress of the Institution.

#### The National Sulphuric Acid Association, Ltd.

THIS Association was founded in 1919 at the recommendation of the Departmental Committee set up to investigate the post-war condition of the Sulphuric Acid and Fertiliser Trades. Its objects include "the promotion of the consideration and discussion of all questions affecting the trade and interests of sulphuric acid manufacturers, and the promotion of efficiency, economy and co-operation in relation to the manufacture and distribution of sulphuric acid." The efficiency of conversion of sulphur in raw material purchased into sulphur in acid manufactured has increased year by year until it has now reached the highly satisfactory figure of over 92 per cent. This percentage is the average applicable to the sulphuric acid industry of the United Kingdom and Ireland, exclusive of acid manufactured as a by-product in metallurgical processes.

TABLE 1.—SULPHURIC ACID.

YEAR.	Acid made, including Oleum in tons of 100 per cent. $H_2SO_4$	Pyrites.		PERCENTAGE MADE FROM		
		Imported.	Domestic.	Spent Oxide.	Brimstone.	Zinc Concentrates.
1924	918,000	48.5	1.0	22.8	23.6	4.1
1925	848,000	45.94	1.1	23.82	23.67	5.57
1926	722,000	46.2	0.92	24.57	23.18	5.13
1927	889,000	42.37	1.16	24.44	24.6	7.42
1928	928,000	44.18	0.81	25.64	20.12	9.25
6 months of 1929	488,000	47.93	1.01	24.23	17.56	9.27

TABLE 2.—PRODUCTION OF SULPHURIC ACID AND CONSUMPTION OF RAW MATERIALS.  
100%  $H_2SO_4$

YEAR.	Acid and Oleum.	Net Acid.	Oleum.	Pyrites.	Spent Oxide.	Sulphur and $H_2S$ .	Zinc Ores.
1924	918,000	874,000	44,000	360,000	152,000	69,700	37,100
1925	848,000	779,000	69,000	313,000	148,000	64,200	57,700
1926	722,000	643,000	79,000	268,000	128,600	53,600	51,300
1927	888,800	773,400	115,400	305,500	155,500	68,700	86,300
1928	928,000	782,000	146,000	329,000	174,000	58,300	116,400
6 months of 1929	488,000	413,000	75,000	185,000	87,000	26,300	58,300

The Association collects, summarises and distributes the only reliable statistics on every phase of the sulphuric acid industry of Great Britain, and these data are available to all members as a guide in estimating their own process and cost efficiency.

The Association during 1929 has continued to carry out the collective purchase of raw material on behalf of certain of its members, with which it has been associated for several years. It also continues to arrange for the marketing, through the agency of its members, of a large proportion of the by-product acid manufactured in metallurgical processes.

The tables at the foot of page 622 give a summary of the sulphuric acid position of the United Kingdom up to June 30, 1929, and it may be added that since this date the progress indicated has been maintained.

### Institution of Petroleum Technologists

THE Institution, of which Dr. A. E. Dunstan was elected president on March 19, was represented at the second International Drilling Congress held in Paris, September 16 to 22, 1929. The British Drilling Committee has, at the request of His Majesty's Government, been formed and the work in connection with it undertaken by the Institution and six papers were forwarded to the Congress. Papers and a complimentary address were sent to the World Engineering Congress held in Tokyo, Japan, in October. Papers were also presented to and representatives attended the ninth Congrès de Chimie Industrielle held at Barcelona in October.

The second edition of the book *Standard Methods of Testing Petroleum and Its Products* was issued in May. The new edition is interleaved with plain pages, and contains several new tests, also many of the original ones have been revised and brought up to date. A new branch of the Institution was formed in South Wales in April, bringing the total number of branches up to four, viz., Roumania, Persia, Trinidad and South Wales. The American Society of Testing Materials has honoured the Institution by appointing the President as Chairman of its Committee on Petroleum Products.

The Redwood Medal for the sessions 1927-28, 1928-29, was awarded to Mr. W. H. Fordham for his paper on Geophysical Surveying, published in *Journal* No. 72 (February, 1929), and the Students' Medal and Prize for the Session 1928-29 was awarded to Mr. H. M. Stanley for his paper on "Recent Advances in our Knowledge of Natural Gas and 'Cracking' Gas, and their Industrial Utilisation." These presentations were made at the annual dinner held on October 11, 1929. The membership to date is 1,171.

### Oil and Colour Chemists' Association

DURING the past year the association has continued to make progress. That the interest in the scientific development of the manufacture of paint and varnish is a lively one is evidenced by the sturdy growth in the membership of this Association. The same rate of increase in the number of the members is noticeable this year as in former years. Progress has also been maintained in the quality and quantity of the papers read before the association, and also in those communicated.

Dr. J. J. Fox, O.B.E., F.I.C., again held the office of president. Many members of the council and of the association continue to serve on the sub-committees and panels of the British Engineering Standards Association. Under the chairmanship of Mr. T. H. Bridge, the Manchester section of the association maintains its steady progress.

At the opening meeting of the new session 1929-30, the president read his address, his subject being "Tests and their Interpretation." As heretofore, the association produced the Section of the Annual Reports on the Progress of Applied Chemistry (1928) dealing with paints, pigments, varnishes and resins; the members concerned with this Section, under the editorship of the honorary editor of the journal of the association, were: Messrs. T. H. Barry, R. R. L. Britton, R. G. Daniels, and W. E. Wornum, the Section being introduced by Mr. C. A. Klein.

The annual dinner was held at the Connaught Rooms, London, on April 16, and on November 29, the Association held its second annual dance.

### The National Benzole Association

DURING the year, the Joint Research Committee of the National Benzole Association and the University of Leeds has continued to pay attention to various problems. The most important piece of research carried out under the direction of the committee has been the continuation and development of the study of resin formation in benzoles. Large scale trials were made to confirm the view, tentatively drawn from laboratory tests, that the addition of "inhibitors" would prevent resinification on storage. Support was obtained from the large scale tests, but more work is to be done before final conclusions are drawn. Another aspect of the same work has dealt with an investigation of the tendency of unrefined benzoles to produce resins on storage.

Wash oils have also been the subject of examination from various points of view—that is, as regards the volatilities and absorptive capacities of oils from low-temperature and Bergius tars for the purpose of benzole recovery; as regards the variation in the absorptive capacities of two typical wash oils, creosote and gas oil, with temperature; and as regards the deterioration of wash oils in use. For the purpose of the preparation of correction tables applicable to hydrometer readings and volumes so as to give the correct value at the standard temperature of 60° F., the densities of mixtures of average samples of benzole and petrol have been determined, and nomograms based on them have been prepared.

### Society of Dyers and Colourists

MEETINGS and lectures under the various sections of the Society have been held in Bradford, Huddersfield, Manchester, London, Leicester, Nottingham, Derby and Glasgow, particulars of which are contained in the January and November Journals.

There has been no further award of the Dyer's Company's Research Medal.

In connection with the "Colour Index" of the Society, the demand for this work still continues and also for the recently published "Supplement" to same.

The work on "Standardising the Methods of Testing the Fastness of Dyed and Coloured Materials" is progressing very favourably, and during the present year a number of Interim Reports have been published.

### Society of Glass Technology

The activities of the Society for the year 1929 included nine meetings, four of which were held in Sheffield, one in Manchester, two in Leeds, and two in London. At these meetings some twenty papers were communicated and discussed. Special mention should be made of the meeting in London in May, which took the form of a joint meeting with the Physical Society, and was combined with visits to the research laboratories of the General Electric Co., Ltd., and to the Osram-G.E.C. Glass Works at Wembley. Members of the Society were also privileged, during the year, to pay visits to the works of the Ford Motor Co., Manchester; the glass works of Beatson, Clark and Co., Ltd., Rotherham; the laboratories of the British Association of Research for the Cocoa, Chocolate, Sugar, Confectionery and Jam Trades, London; the works of Hadfields, Ltd., Sheffield; and the quarries of Yorkshire Amalgamated Products, Ltd., Warmsworth.

A notable event in the history of the Society occurred on the occasion of the general meeting held in London on May 15, when a London Section was formally inaugurated, in order to cater for the needs of members who were generally unable to attend the ordinary meetings of the Society, which are usually held in the afternoon. Consequently, it was decided to hold meetings monthly in the evening, and to combine with the meetings, where possible, visits to works or laboratories. The first meeting of the London section under the new scheme was held on October 2, and was well attended. Mr. Verney Stott, of the National Physical Laboratory, Teddington, was appointed honorary secretary to the section.

In addition to the issue quarterly of the *Journal*, there were also published during the year:—*Report of the First British Glass Convention held at Bournemouth, September 10-22, 1928*. This report ran to 96 pages and was issued jointly by the Society of Glass Technology and the Glass Manufacturers' Federation,

## The I.C.I. Dyestuffs Patent Action

### Views of The Comptroller of Patents

*The following is an account of the latest proceedings in the patent action between I.C.I. and the I.G., now taking place before Mr. Justice Maugham. Reports of earlier proceedings appeared on November 23 and 30, and December 7, 14, and 21.*

On Thursday, December 19, Mr. Frank Sharples, inside works manager of Chadwick and Co., of Oldham, dyers, bleachers and finishers of cotton piece goods, said that his firm dealt with some 70 or 80 tons of cotton goods a week. About 25 per cent. of these were grey and with coloured threads. They bleached and kier boiled or kier boiled samples to show the fastness. Witness was handed a sample book of dyed hanks which had been kier boiled, and he said that from a commercial point of view he would only pass two out of the seven samples.

His Lordship enquired if witness had had any experience of firms asking for a little money because goods were not up to sample.

#### The Result of the Tests

Witness said that samples of goods to be dyed were submitted to their laboratory, and if they found any change of tone on a test, the firm were notified, and if the firm took the risk of having the goods dyed, these were put through the usual processes. If they refused to take the risk, then they sent the goods back.

His Lordship: Is there any provision for arbitration in case of dispute?

Witness said that he believed that in case of any dispute a Committee of the Chamber of Commerce dealt with the matter. Replying to a further question by his Lordship, witness said he often had to judge samples of colour dyeing by small hank samples, as well as samples a yard square.

His Lordship: Then a sample the size of a penny stamp is enough for you?—Yes.

This closed the petitioners' case.

Mr. Whitehead said he would call Mr. Lawson to continue his evidence on Friday.

His Lordship said that under those circumstances, he would now hear Sir Arthur Colefax, K.C., for the Comptroller of Patents, and after the evidence of Mr. Lawson would adjourn the hearing of the case until January 14.

#### The Position of the Comptroller

Sir Arthur Colefax said that the Comptroller had nothing to do with the dispute between the parties in this legislation; all he had to consider was the question of the proposed amendments, and to look at them from the standpoint of safeguarding the interests of the public. He would submit to the Court the views of the Comptroller with respect to the particular amendments sought, and how far they should, within his Lordship's powers, be allowed within the discretionary power conferred. He would also have to deal with Section 32a, which his Lordship had referred to at the end of Mr. Cripps' speech on the previous Friday.

Dealing with the question of the permissibility of these amendments, whether or not it would make the invention claimed substantially different from, and substantially larger than what was previously claimed, Sir Arthur said that the Comptroller took this view: That the overriding consideration was the patent granted in 1912, viz., No. 6,379, and granted to the owners of the specifications sought to be amended. The Comptroller took the view that one must have regard to the 1912 patent in order to see what, if any, was the scope of valid subject matter in the application of later date.

#### The Effect of the Amendments

The first patent sought to be amended was 199,771, and he submitted that in the amendment the selection was different from that for which the patent was granted. This was a case where amendments would make the invention claimed substantially different from that claimed in the specification as it stood. The amendment cut out a large number of diazo bodies from those selected, with which the original specification was concerned, and cut out the diazo contained in the nitro group. A large number of couplings were also cut out.

His contention was that here, in the opinion of the Comptroller, the respondents were seeking to alter the selection and make a different selection. Would the specification as amended be a claim to the discovery made in the specification

or patent? In the view of the Comptroller it clearly would not, but would be something quite different.

In regard to the point of the exercise of discretion by his Lordship, Sir Arthur cited a number of decisions, and said that the Court ought to consider the line of conduct of the patentee. If he put in a broad claim to warn off the public and to check development of research and the progress of industry, and then at a later date, many years after the patent, and then only in response to proceedings for revocation, sought to amend—these were matters his Lordship could properly take into account in considering whether or not his discretion should be exercised.

Dealing with Section 32a, which gave the Court power to give relief under certain circumstances, he submitted that that section should be read with an earlier section, and that it merely meant that the Court had power to grant relief if it were satisfied that the conditions laid down in the section applied.

This concluded the speech for the Comptroller.

On Friday, December 20, Mr. Lawson was recalled for respondents, and the hearing was adjourned to January 14, when counsel will address the Court. The speeches are expected to last four days.

## The Claude Process

### Some Recent Developments

THE total production of all synthetic ammonia plants using the Georges Claude process has grown considerably, and a further progress is to be expected, since new plants have been established in the course of the current year. The Société Chimique de la Grande Paroisse is building an important plant for the application of this process at Grand Quevilly, near Rouen, and has concluded an agreement with the Compagnie de Saint-Gobain for placing the output of this plant. The Société l'Ammoniaque Synthétique, working with licences of the Société Chimique de la Grande Paroisse, has established a plant with the assistance of the Compagnie des Mines d'Aniche, and with the recent installation of new apparatus has doubled its production.

The Société Chimique de la Grande Paroisse has, in combination with the Société Houillère de Lievin, established the new company Ammoniaque de Lievin, which holds a licence for the production of synthetic ammonia with gas from coke ovens. The Société Belge de l'Azote, using the same licences, has carried on its programme of extension and has greatly increased its capacity of production. The results obtained in the plant of the Société Tchèque des Produits Azotes are very satisfactory, and further improvements in production are contemplated. The Société Fermière des Mines Fiscales, of Poland, has just completed, at its plant at Knurow, the installation of a new unit which will permit of a considerable increase in production.

## "Buy British" in America

AN interesting and very important plea to British shipowners passing through the Panama Canal is made by the F.B.I. representative at Panama.

No less than 30 per cent. of the ships using the Canal are British, and practically every ship that does so buys stores from the commissaries of the Canal Zone—i.e., United States Government. Naturally these commissaries stock U.S. goods by preference, though their real policy is stated to be to try and supply anything they are asked for. The Federation's representative urges that the owners of British ships using the Canal should make persistent requests, when ordering goods, that such goods should so far as possible be of British origin. He states that he is emphatically of the opinion that if steamship owners would only persist in asking for British goods the commissaries would keep these in stock and that then the superior quality of the British article would probably definitely guarantee it increased sales.

## Bromine Recovery in Alsace

### Growth of the French Industry

THE French bromine industry in Alsace is only of comparatively recent date. During the war, the bromine necessary for the production of poison gas for the French army was obtained from America, which, next to Germany, is the largest producer of bromine. Prior to the war, the French bromine requirements were almost entirely imported from Germany.

During the war a bromine recovery plant was erected near the mouth of the Rhone in Southern France, and another plant was erected in Tunis, 150 kilometres from Zarzil, where the waters of the salt lake which contain up to 6 kg. of bromine per cubic metre were evaporated. The production of this plant amounted to about two tons daily. At present this plant is not in active operation, although it is maintained in view of possible future military requirements. The plant at the Rhone mouth belonging to the Société d'Alais Froges et Carmargue recovers bromine to the extent of about 125 tons yearly.

As lately as 1926 France imported considerable quantities of bromine from Germany. In 1925, the imports were 34 tons of bromine and 33 tons of bromine salts; in 1926, 50 tons of bromine and 27 tons of bromine salts.

### The Beginning

In 1921, bromine recovery towers, with a capacity of 60 tons of bromine yearly, were installed at the Alsatian potash mines, and in 1927 imports of bromine from Germany ceased entirely. The first of the bromine recovery plants was installed by the Société Amalle, and this came into operation in July, 1926. Since that date, other plants have been erected at the same place.

A bromine recovery plant has also been erected and is being operated at the St. Thérèse Potash Mines (near the potassium chloride factories in Ensisheim), which has a capacity of 180 tons of bromine yearly. In 1928 the Amalle Société treated 58,340 cubic metres of mother liquor, compared with 33,000 cubic metres in 1927, and obtained 111,818 kg. of bromine against 63,494 kg. in the previous year. In the Ensisheim factory in 1928, 46,152 cubic metres of mother liquor were treated against 14,000 cubic metres in the previous year, and the bromine recoveries in the respective years were 69,330 kg. and 20,007 kg. It will be seen, therefore, that the Alsace bromine industry is an expanding one, limited, of course, by the market possibilities.

### Bromine Content of Alsatian Potash

The bromine content of the crude potassium salts in Alsace is relatively high, but varies according to the content of pure potash. The rock-salt beds there contain on an average only 0.03 per cent. of bromine, but the sylvinite as mined contains up to 0.35 per cent.; it is found that the more potash there is contained in the crude sylvinite, the larger is the percentage of bromine. On an average, however, the extracted salts contain not more than 1 per cent. of bromine.

The bromine recovery process used in Alsace is connected with the production of potassium chloride. The crude salts containing from 16 to 20 per cent. of  $K_2O$  are first treated in the mother liquor from a previous crystallising process, at a temperature of round about  $106^{\circ}C$ . Under these conditions, the potassium chloride in the crude salt becomes dissolved in this mother liquor, while the sodium chloride content in the crude salt is only very slightly dissolved and remains in the dissolving tanks. The saturated liquor is clarified and cooled, and potassium chloride of 75 per cent. purity crystallises out. The mother liquor from this crystallisation can be used for dissolving a further quantity of potassium chloride. The crude bromides in the raw salt are also dissolved in the mother liquor. This liquor becomes rather rich in bromine, but never saturated.

### Method of Treatment

The potassium chloride factory of the Société Theodor treats a 22 per cent. salt with a bromine content of about 0.12 per cent. The mother liquor contains on an average 3.8 grams per litre. The bromine is recovered from the mother liquor, which can be used again in the production of potassium chloride after neutralisation. However, from practical considerations, the bromine content of this liquor is never reduced below 2 grams per litre.

For obtaining bromine from the mother liquor at the Alsace Potash Mines, the Kubierschky tower is used. The mother liquor, heated to  $75^{\circ}C$ ., is brought into contact with chlorine gas in this tower. The bromide is expelled as vapour, which is collected by condensation. It is then heated to drive out the remains of chlorine, so that the bromine finally contains not more than 0.2 per cent. of chlorine. The Kubierschky tower has a capacity for treating 80 cubic metres of mother liquor daily, which corresponds to a daily production of about 200 kg. of bromine.

The total French bromine production, including that of the Société d'Alais Froges et Carmargue, was about 300 tons in 1928, but this quantity could be increased very greatly if the full possibilities of the Alsace potash industry were utilised. Whether this will happen in the future depends upon the market possibilities, as the French requirements are only about 200 tons a year. If the market for bromine can be expanded by new uses for it, it is certain that new recovery plants will be erected in Alsace.

## Food and Drugs Acts

### Annual Report of Ministry of Health

A PAMPHLET under the title "Sale of Food and Drugs Acts (Extracts from the Annual Report of the Ministry of Health for 1928-29 and Abstracts of Reports of Public Analysts for the year 1928)" has just been published by H.M. Stationery Office (pp. 15, 1s. 6d.)

129,034 samples of food and drugs were reported upon by Public Analysts in England and Wales in 1928, an increase of 4,770 over 1927. 7,524 were reported as adulterated or not up to standard, a proportion of 5.8 per cent., the same as in 1926, and slightly more than the proportion (5.5 per cent.) for 1927. 757 contraventions of the Public Health (Preservatives, etc., in Food) Regulations were reported, which may be regarded as a comparatively small number since, except as regards manufactured articles of food containing butter, the Regulations were fully operative throughout the year. The Regulations applied to manufactured articles containing butter from July 1, 1928.

### Milk, Cream, Butter, etc.

67,350 samples of milk, being more than half the total number of samples of all foods, were examined. Of these 5,542 or 8.2 per cent., were reported against as adulterated or not up to standard, comparing with 7.4 per cent. in 1926 and 6.9 per cent. in 1927. It is a matter for speculation how far, if at all, this increase represents a real increase in the practice of adulteration and how far it is accounted for by the increasing tendency of Local Authorities to concentrate their sampling on suspected sources.

1928 was the first year in which the sale of cream containing preservatives was prohibited. 2,671 samples of cream were examined, of which 70 were reported against, 55 as containing boron preservative, 10 as being deficient in fat or containing excess water, and 5 as being reconstituted or artificial cream.

10,544 samples of butter were examined, of which 264, or 2.5 per cent., were reported against. This is a much higher proportion than that reported for several years past, the difference being due to the operation of the Preservatives Regulations. The proportion of samples reported against for reasons other than presence of preservatives was about the same as in previous years.

Copper added for colouring purposes was found in one sample of tinned peas and in one of tinned beans, and two samples of pickles were reported to be slightly contaminated with that metal. Zinc was found in one sample of cider and also in one of ice cream. Tin was found in 25 samples of tinned fruit, tinned fish and tinned soup.

### Drugs

4,793 samples representing 131 different kinds of drugs were examined, and 262, or 5.6 per cent., were found to be adulterated as compared with 5.7 per cent. in 1927. Proceedings were successfully instituted in respect of twenty out of the thirty prescriptions which on analysis proved to have been incorrectly dispensed, or prepared from drugs not of the nature prescribed. Three samples of almond oil were reported to be mainly composed of peach or apricot kernel oil.

## Scottish Coal Tar Products During 1929

(FROM OUR SCOTTISH CORRESPONDENT.)

CONDITIONS throughout the year have not been entirely up to expectations, but nevertheless values generally are on a higher level at the present time than they were twelve months ago. Creosote oil has been the subject of much thought and, although the position is still far from satisfactory, it is pleasing to note that steps are being taken to find new uses for it and also to further the present main use, namely, timber preserving. The cause of the slump in creosote is, of course, the fact that America is not purchasing such large quantities owing to her domestic production having been increased within recent years. The position, however, has been helped greatly by the steady demand for cresylic acids of all grades, with the exception perhaps of high boiling cresylic which has not been particularly active throughout the year.

**Cresylic Acid.**—Taking pale 97/99% acid as standard, the lowest average quoted price during the year was 1s. 7½d. per gallon and the highest 2s. per gallon. The value during January was about 1s. 8½d. per gallon which gradually depreciated to about 1s. 7½d. per gallon in July, when it rose steadily to about 2s. per gallon in November.

**Carbolic Sixties.**—A graph curve showing the trend of this article would be very similar to one of cresylic acid. Lowest price was about 1s. 11d. per gallon and highest about 2s. 9d. per gallon, with the apex during October.

**Creosote Oil.**—This article has decreased in value from about 5d. per gallon in January to 3d. per gallon in August, rising again to about 4d. per gallon in December. Specification oil is taken as standard.

**Coal Tar Pitch.**—Manufacturers have been in closer touch with each other than hitherto. Prices are more under control, but pitch being probably one of the most speculative articles in the by-products group, the value is primarily fixed by the demand from South Wales and Continental briquette makers. The tendency this year has been upwards, starting in January at about 37s. 6d. per ton and rising to to-day's value of about 50s. per ton. The rise, however, has not been the usual one associated with pitch, as it has taken the full twelve months to gain about 12s. 6d. per ton. June and July were the interesting months with pitch, as it was then that value jumped from about 37s. 6d. per ton to 45s. per ton.

**Blast Furnace Pitch.**—The production this year has been greater than the demand and the ironmasters are at present over-stocked. Price fell rapidly from about 27s. 6d. per ton in January to about 17s. 6d. per ton in May, when it gradually regained its position. In September the price was fixed by mutual agreement among the ironmasters at to-day's quotation of 30s. per ton, f.o.r. works. This article invariably follows closely on the heels of coal tar pitch.

**Refined Coal Tar.**—The trend of prices this year has been a reverse of the usual and of what is generally anticipated. Starting in January at 3½d. per gallon value gradually fell to a fraction under 3d. per gallon in May, at which figure it remained during the road tarring season. The value then rose to to-day's figure of about 4d. per gallon. Surveyors, contractors, etc., obtained their supplies at lower prices than have existed since pre-war days. It is generally anticipated, however, that they will not be so fortunate during the coming season, and already some of the large contractors have placed orders for forward delivery at prices from 3½d. per gallon upwards, according to quantity, quality and district.

**Blast Furnace Tar.**—Business throughout the year has been very disappointing indeed. From 3d. per gallon in January, value dropped to 2d. per gallon in March, at which figure it remained until the ironmasters fixed the price by agreement at 2½d. per gallon in September. Continental contractors use considerable quantities for road making but in this country the outlet is limited.

**Crude Naphtha** benefited by the import duty on foreign spirits which became effective early in March. Prices throughout the twelve months under review have remained fairly constant, but in Scotland the production is so comparatively small that values fluctuate more or less according to the pro-

duction in England. The average price over the year was about 4½d. to 5d. per gallon.

**Water White Products.**—The tax on foreign spirit was of considerable benefit to refiners, but prices did not advance by the full 4d. per gallon. Naphtha only rose about 1d. per gallon and motor benzole about 2d. per gallon. It is probably safe to say that the oil companies did not lose anything by the imposition of the petrol tax. On the contrary it is probable that they have gained. The price of petrol was increased by 4d. per gallon to the consumer, but the oil companies, who are the largest buyers of coal tar spirits, were only paying about 1d. to 2d. per gallon extra for naphthas and benzole. 90/160 solvent was about 1s. 1d. per gallon in January and rose in March to 1s. 2d. per gallon. Value then rose gradually to round 1s. 4½d. to 1s. 5d. per gallon in May, when it sagged to to-day's level of 1s. 1d. to 1s. 2d. per gallon. Motor benzole moved at approximately the same time as naphtha and in the same direction. From 1s. 5½d. per gallon in January and 1s. 7½d. per gallon after imposition of tax in March, value remained fairly steady until July when it gradually decreased to 1s. 5d. to 1s. 5½d. per gallon, at which level it now stands.

## Chemistry and Motor Cycles

By G. L. Hack

(CHIEF CHEMIST AND DEVELOPMENT ENGINEER, RUDGE-WHITWORTH, LTD.).

ALTHOUGH every large engineering factory now has its laboratory, not many firms can claim that a laboratory has been an integral part of their organisation since 1900, which was the year in which Rudge-Whitworth, Ltd., equipped a department in the heart of their works which was promptly termed "Stinks" or the "Chemist's Shop" by the employees. Starting in a very small way with investigations into some of the problems met with in the nickel-plating process of that time, the work of the laboratory has gradually increased until it now conducts development work of all kinds and controls various works processes. While much of its work necessarily consists of physical testing and investigations of a purely physical character, the chemical section of the laboratory is a most important one.

### Testing and Analytical Work

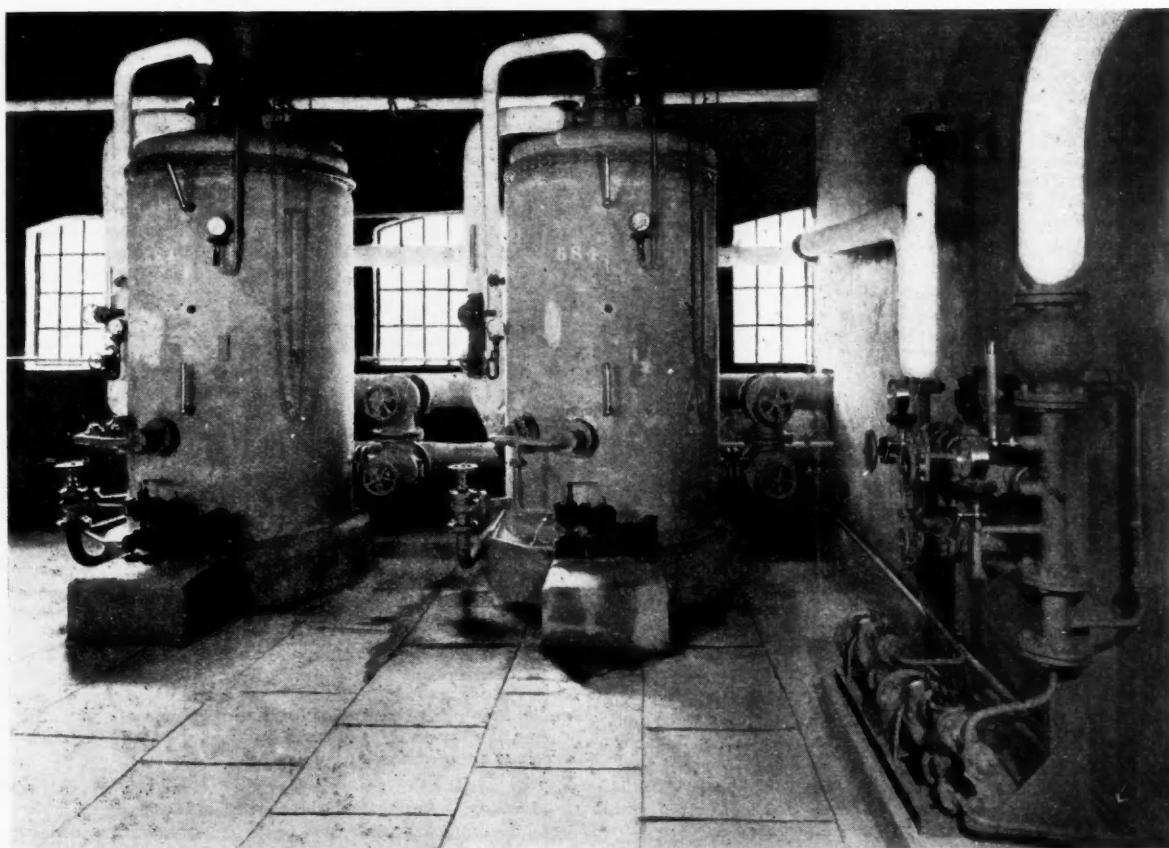
Steel, bronzes, aluminium alloys and brazing brasses are regularly analysed to ensure that only materials which conform to the specification to which they are ordered reach the machine shops. All plating vat solutions are tested weekly. A warm solution containing sodium chloride and boric acid is used for nickel deposits, the density and pH value providing adequate data for proper control. Copper deposits are chiefly required for protecting portions of components during the carburising process, and a solution of the double cyanide type is employed. This is analysed for copper and total cyanide. A large-scale chromium plating plant is a comparatively recent installation, but for the previous twelve months two small experimental vats were kept in continuous operation in the laboratory, analyses being frequently made. The work has been amply repaid by the fact that the large-scale plant has been operated without trouble, and density and sulphate-chromium ratio determinations are all that have been found necessary to enable the vat to be kept in proper condition.

Gas analysis is also carried out on occasion. Knowledge of the composition of hardening-furnace flue-gas can often result in fuel economy, and the analyses of exhaust gases from motor-cycle engines on bench test often reveal valuable data in the search for higher efficiencies.

### Unemployment Statistics

THE number of insured persons recorded as unemployed at November 25 in the chemical industry in Great Britain was 7,114 (males 6,392, females 722); in the explosives industry 1,055 (males 776, females 279); in the paint, varnish, japan, red and white lead industries, 893 (males 714, females 179); and in the oil, grease, glue, soap, ink, match, etc., industries, 4,859 (males 3,936, females 923). The percentages unemployed (of the total numbers insured) in the same industries in the same order were 6·8, 5·7, 4·7 and 6·5 respectively.

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## Patent Literature

The following information is prepared from published Patent Specifications and from the Illustrated Official Journal (Patents) by permission of the Controller to H.M. Stationery Office. Printed copies of full Patent Specifications accepted may be obtained from the Patent Office, 25, Southampton Buildings, London, W.C.2, at 1s. each.

### Abstracts of Accepted Specifications

320,842. CELLULOSE ESTERS. H. Dreyfus, 22, Hanover Square, London. Application date, April 18, 1928.

Halogen-substituted cellulose derivatives are obtained by acylating cellulose materials with chlor- or brom-aliphatic anhydrides, e.g., the anhydrides of chlor- or brom-acetic acid or  $\alpha$ - or  $\beta$ -chlor- or brom-propionic acid. Mixed esters can be obtained by employing another anhydride such as acetic anhydride or its homologues, in addition to the halogen aliphatic anhydride. The cellulosic materials may be subjected to a preliminary treatment with formic or acetic acids, or with hydrochloric acid. The acylation may be conducted in the presence of a catalyst such as sulphuric acid, benzene-sulphonic acid, bi-sulphates, zinc chloride, phosphoric acid, or sulphuryl chloride. When hydrochloric acid is used, it may be employed in conjunction with chlorides of iron, tin, manganese, copper, nickel, or cobalt. The acylation may be effected in the presence of solvents for the cellulose derivative produced. The halogen-substituted cellulose derivatives may be converted into other derivatives by wholly or partly replacing the halogen by treating with hydrolysing agents, such as caustic soda solution. The hydroxy-containing derivatives may be further esterified to obtain acyl-oxy-alkacyl derivatives, or mixed alkacyl-acyl-oxy-alkacyl derivatives. The halogen-substituted cellulose derivatives may be converted into other derivatives by treating with ammonia or amines such as methyl-amine, dimethyl-amine, ethylamine, dimethylamine, aniline, or methyl or ethyl-aniline. Examples of all these derivatives are given.

320,845. DECOMPOSING ORES. R. W. Stimson, 17, West 56th Street, New York. Application date, April 21, 1928.

A granulated or powdered ore or other material containing chromium, manganese, molybdenum, titanium, uranium, or other metals having acidic oxides is mixed with a base or salt such as sodium nitrate, and heated to 700° C. in the presence of nitrogen tetroxide. Nitric oxide is liberated, and may be cooled, purified, and converted into the tetroxide for use again. The initial charge may consist of a mixture of ore and sodium or potassium nitrate, and subsequent charges may consist of ore and sodium carbonate. The products may be leached with water or dilute sulphuric acid, bichromates being obtained in the latter case when a chromium ore is treated. Alternatively, hot water at different temperatures may be used to obtain chromate and bichromate. The granular ferric oxide residue may be briquetted, and used for the production of iron. The alkali bichromate solution obtained may be mixed with lime, evaporated, and fused with ferro-chromium-silicon alloy to obtain a rich ferro-chromium.

320,846. HYDROCARBONS. J. Y. Johnson, London. From I.G. Farbenindustrie Akt.-Ges., Frankfurt-on-Main, Germany. Application date, May 18, 1928.

The hydrocarbons obtained by the destructive hydrogenation of coal, oil, shale, etc., or by cracking oils and tars, or by low-temperature carbonisation of coal, are converted into products of higher boiling point and increased viscosity by treating them with halogens or compounds yielding halogen, with the addition of an element from groups 3—8 which is capable of existing in metallic form, or compounds of mixtures of these. The halogens or halogen compounds may then be removed and the products further condensed. If the initial materials contain a considerable proportion of aromatic hydrocarbons, lubricating oils of the nature of cylinder oils are obtained. The halogen-exchanging substances may be methylene chloride, chloroform, or carbon tetra-chloride or they may be obtained by treating the starting material with halogen and catalysts such as aluminium chloride, tin chloride, iron chloride, boron fluoride, phosphorus pentachloride, silicon tetrachloride, antimony chloride, chromium chloride, phosphorus pentoxide, or alumina, or elements such as aluminium, iron, boron, tin, antimony, vanadium, chromium, molybdenum, or tungsten. Several other alternatives and modifications are described. Examples are given of the treatment of middle oils from petroleum and brown coal,

heavy benzines, chlorinated heavy benzines obtained from various sources, and crude naphthalene.

320,891. TREATING OLEIFEROUS RESIDUES. J. Y. Johnson, London. From I.G. Farbenindustrie Akt.-Ges., Frankfurt-on-Main, Germany. Application date, July 23, 1928.

This process is for treating residues obtained in the treatment of mineral oils, tar oils, or olefines, with metal halides, or with iron or aluminium and halogen hydride, or the residues obtained by refining hydrocarbon oils with sulphuric acid. These substances are subjected to dry distillation above 360° C. with the addition of calcium carbonate or other basic salts, Fuller's earth, lead salts or oxides, iron oxide, powdered quicklime, or calcined magnesite. The first runnings from the distillation residues from refining hydrocarbons with sulphuric acid may be treated to obtain lubricating oils by polymerisation and condensation. An example is given.

320,918. DESTRUCTIVE HYDROGENATION. J. Y. Johnson, London. From I.G. Farbenindustrie Akt.-Ges., Frankfurt-on-Main, Germany. Application date, May 21, 1928.

Carbonaceous materials are subjected to a preliminary treatment by distilling off water in the presence of an organic solvent such as mineral and tar oils of high boiling point, naphthalene, anthracene, etc. The distillation may be effected at reduced pressure or in the presence of scavenging gases. High pressures may also be used, and the process may be effected in several stages at 300°–450° C. An example is given of the treatment of brown coal before its destructive hydrogenation, or pressure treatment with solvents.

320,921. PURIFYING WAXES. J. Y. Johnson, London. From I.G. Farbenindustrie Akt.-Ges., Frankfurt-on-Main, Germany. Application date, July 19, 1928.

Crude paraffin wax is treated at a temperature of 300°–400° C. and pressure of 10–250 atmospheres with hydrogen or gases containing it, the conditions being such that little or no cracking takes place. A catalyst may be employed consisting of one or more oxides of the metals of groups 3, 4 or 6, together with iron, nickel, or cobalt, or their oxides as activators. Suitable catalysts include molybdic acid, mixtures of molybdenum trioxide, or dioxide with alumina, nickel oxide, zirconium oxide, chromic oxide, silica gel, or cerium oxide, or a mixture of aluminium and chromic oxides, or a mixture of zirconium oxide and chromic acid. Examples are given.

320,930. CATALYTIC OXIDATION OF SULPHUROUS GASES. S. Robson, The Bungalow, St. Andrews Road, Avonmouth. Application date, July 25, 1928.

Sulphur dioxide and other sulphurous gases are mixed with air or oxygen and passed over iron oxide in a sintered or porous nodular form. The catalyst may be mixed with smaller amounts of oxides of chromium, manganese, vanadium, or zinc, and may be obtained by blast roasting pyrites as described in specification 307,439.

320,937. MAGNESIUM CARBONATE. T. Hughes, 265, Strand, London. Application date, July 26, 1928.

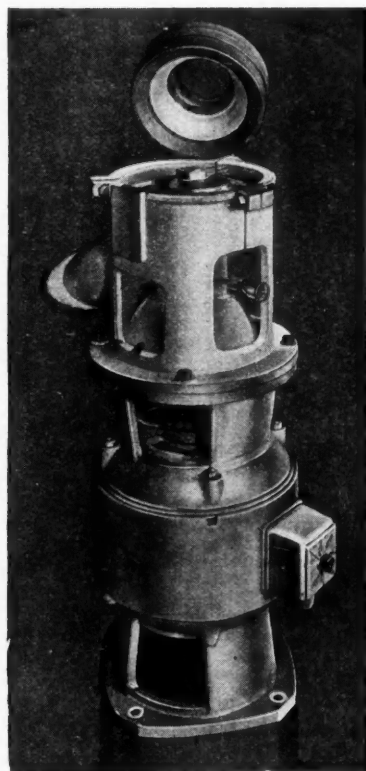
Magnesium carbonate and other compounds are obtained from dense hard varieties of magnesite, which remain dense after successive treatment with small amounts of dilute mineral acid and contain basic and non-basic carbonates and also iron compounds, silicates, and calcium compounds. The raw material is treated with mineral acid in such a way as to permit selective action on the constituents and prevent substantial disintegration.

Magnesite in large lumps is treated at 60° F. for 4–5 hours with dilute sulphuric acid, and the acid solution of magnesium sulphate with normal magnesium carbonate in suspension is circulated to a high-level terrace cascade of separator tanks. The magnesium carbonate settles out, and the liquid from the lowest tank is delivered to the reaction vessel. After the reaction is complete, the products are passed through a second group of separator tanks, and the normal magnesium carbonate washed and dried. It is suitable for paints, fireproof compositions, etc. Permanent suspensions can be made in a colloid mill without a stabilizing agent.

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**PREMIER COLLOID  
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320,960. SYNTHETIC RUBBER. J. Y. Johnson, London. From I.G. Farbenindustrie Akt.-Ges., Frankfurt-on-Main, Germany. Application date, April 26, 1928.

Emulsions of diolefines are polymerized in the presence of hydrogen peroxide and Carragheen moss extract, gum arabic, molasses or linseed meal extract, with or without substances favouring polymerization, such as emulsifying agents, substances affecting the surface tension such as amyl, heptyl, benzyl, etc., alcohols, electrolytes, and buffer substances. Thus, in one example, an emulsion of isoprene, linseed meal extract in skim milk, and hydrogen peroxide is heated to 75° C. for 2-3 days, and in another example, an emulsion of isoprene, Carragheen moss extract, sodium isopropyl-3-naphthalene sulphonate, glue, and hydrogen peroxide is heated to 75° C. for 2 days.

NOTE.—Abstracts of the following specifications which are now accepted, appeared in THE CHEMICAL AGE when they became open to inspection under the International Convention:—297,777 (Roessler and Hasslacher Chemical Co.), relating to production of perborates, see Vol. XIX, p. 519; 298,611 (I.G. Farbenindustrie Akt.-Ges.), relating to denaturing alcohol, see Vol. XIX, p. 565; 298,494 (Soc. of Chemical Industry in Basle), relating to dyestuffs containing metal, see Vol. XIX, p. 565; 301,734 (British Celanese, Ltd.), relating to concentration of lower aliphatic acids, see Vol. XX, p. 130; 302,599 (I.G. Farbenindustrie Akt.-Ges.), relating to azo dyestuffs insoluble in water, see Vol. XX, p. 189; 303,066 (F. E. Lathé), relating to refining of nickel-copper matte, see Vol. XX, p. 31 (Metallurgical Section); 303,374 (J. P. P. Mayor), relating to manufacture of catalysts, see Vol. XX, p. 234; 304,282 (G. A. Favre), relating to production of anhydrous stannic chloride from metals containing tin, see Vol. XX, p. 283; 305,136 (F. Bensa), relating to vat dyestuffs, see Vol. XX, p. 340.

#### Specifications Accepted with Date of Application

- 291,461. Manures or fertilisers. E. J. Burban. June 2, 1928.  
 296,079. Fatty acids free or almost free from unsaponifiable matter, Method of obtaining. Oel-und Fett-Chemie Ges. August 26, 1927.  
 296,309. N-oxethyl-derivatives of 2-amino-1-oxybenzene, Manufacture of. I.G. Farbenindustrie Akt.-Ges. August 26, 1927. Addition to 280,873.  
 300,964. Benzene hydrocarbons from distillation gases, Method of extracting. C. Still. November 21, 1927. Addition to 293,702.  
 301,898. Divinyl and homologues thereof, Manufacture of. I.G. Farbenindustrie Akt.-Ges. December 9, 1927. Addition to 291,748.  
 306,447. Azo-dyes, Production of conversion products of. J. R. Geigy Soc. Anon. February 20, 1928.  
 306,939. N-methyl-p-aminophenol, Manufacture of. Chemische Fabrik Grünau, Landshoff, and Meyer Akt.-Ges. February 29, 1928.  
 307,889. Distillation of finely divided coal or similar material. Trocknungs-, Verschmelzungs- und Vergasungs-Ges. March 15, 1928.  
 309,161. Rubber, Method of preserving. Goodyear Tire and Rubber Co. April 7, 1928.  
 310,949. Titanium dioxide hydrate from hydrolysable solutions of titanium salts, Separation of. J. Blumenfeld. May 5, 1928.  
 322,427. Finely divided nitroguanidine and like bodies, Preparation of. Nobel Industries, Ltd. (E. I. Du Pont de Nemours and Co.) June 1, 1928.  
 322,437. Unsaturated fatty acids, Manufacture of. J. Y. Johnson. (I.G. Farbenindustrie Akt.-Ges.) August 30, 1928.  
 322,445. Hydroaromatic hydrocarbons, Manufacture of. J. Y. Johnson. (I.G. Farbenindustrie Akt.-Ges.) September 3, 1928.  
 322,446. Alloys of iron and aluminium, Manufacture of. C. P. Sandberg, O. F. A. Sandberg, and N. P. P. Sandberg (trading as Sandberg), and J. W. Bamfylde. September 3, 1928.  
 322,465. Valuable products from organised substances, Production of. J. Y. Johnson. (I.G. Farbenindustrie Akt.-Ges.) June 2, 1928.  
 322,471. Gases and vapours, Treatment of—with electric arc discharges. J. Y. Johnson. (I.G. Farbenindustrie Akt.-Ges.) August 29, 1928.  
 322,489. Valuable products from coal, tars, mineral oils, and the like, Recovery of. J. Y. Johnson. (I.G. Farbenindustrie Akt.-Ges.) September 3, 1928.  
 32,524. Olefines, Manufacture of. A. Carpmæl. (I.G. Farbenindustrie Akt.-Ges.) September 7, 1928.  
 322,527. Copper ores, Concentration of. S. Tucker. September 7, 1928.

322,576. Anthraquinone derivatives, Manufacture of. Imperial Chemical Industries, Ltd., F. Lodge and W. W. Tatum. October 2, 1928.

322,700. Ammonium phosphates, Production of. J. Guillissen, and Union Chimique Belge Soc. Anon. February 22, 1929.

322,701. Synthesis of ammonia, Manufacture of mixtures of nitrogen and hydrogen for. J. Y. Johnson. (I.G. Farbenindustrie Akt.-Ges.) February 22, 1929. Addition to 307,529.

## Research on Iodine

### Work of the Mellon Institute

SINCE January 1, 1928, the Mellon Institute of Industrial Research, Pittsburgh, Pa., has had in operation a Multiple Industrial Fellowship founded for the purpose of investigating the properties and uses of iodine. This Fellowship, which is sustained by the Iodine Educational Bureau, of 64, Water Street, New York, is headed by Dr. George M. Karns, formerly a member of the chemical faculty of the University of Illinois. All results of the Fellowship studies will be published.

### Vehicles and Solvents for Iodine

Recently, through an additional appropriation from the Fellowship donor, the Mellon Institute, acting for the Iodine Fellowship, has made arrangements for the study of certain iodine problems in other institutions that have special facilities for such types of work. A scholarship has been founded at the Philadelphia College of Pharmacy and Science by a research grant from the Institute. This scholarship, which will be held by Mr. L. F. Tice, will have for its aim a broad investigation of vehicles and solvents for iodine, especially for external use in medicine. A large number of new organic chemicals will be studied as solvents with the object of evolving, if possible, a more satisfactory preparation than the alcoholic tincture now in use. The research, for which a definite programme has been laid down, will be supervised by Professor Charles H. LaWall with the advisory collaboration of other faculty members of the Philadelphia College of Pharmacy and Science and with the direct co-operation of Dr. Karns. The investigational findings of the scholarship will be reported in the literature.

### Nutritional Value of Iodine for Live Stock

Another phase of the research programme includes a grant made to the Pennsylvania State College for a comprehensive investigation—under the direction of Professor E. B. Forbes of the Institute of Animal Nutrition—of the nutritional place and value of iodine in the feeding of live stock. Despite the large amount of work which has been done on the rôle of iodine in metabolism, especially with reference to the thyroid, very little is known regarding the specific dietetic aspects of this element, particularly in the lower animals. Dr. Karns and his co-workers on the Iodine Fellowship of the Mellon Institute are co-operating closely with Dr. Forbes and his staff, mainly by preparing standardised feeds. The findings of this research also will be made available to the public, in accordance with the Iodine Educational Bureau's policy of disseminating to every one interested the results of all investigations made under its ægis.

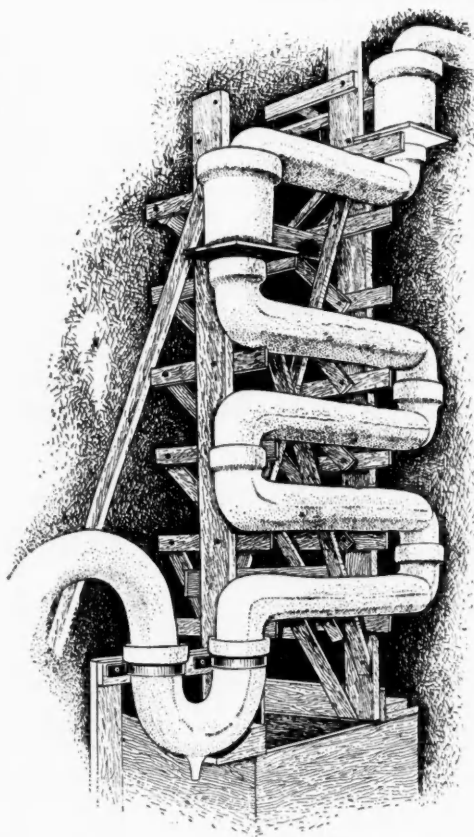
The Mellon Institute is giving consideration to the founding of a research scholarship in a medical school for the purpose of aiding in the solution of incompletely answered questions respecting the utility of iodine in internal medicine. A number of pharmacologists are aiding the Institute in determining a programme for such pharmacodynamic inquiry.

### Carbide Market in Singapore

THE carbide market in Singapore is estimated as fairly large, but no figures of imports are available. The principal industrial activities in this region are rubber and sugar production and oil refining. In the principal cities, such as Singapore, machine and auto repair shops account for some of the carbide consumption. In Java and the outer provinces of the Netherlands East Indies, however, statistics covering 1927 show a total importation of carbide valued at about \$152,166. Approximately 75 per cent. of this total was imported into Java; 226,900 kilos went to Borneo; 51,315 to East Coast of Sumatra; 75,474 to West Coast of Sumatra; 49,749 to Palembang, and 10,316 to Haven Velawan.

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## Chemicals for South Africa

(FROM A CORRESPONDENT.)

WITH South Africa's industrial expansion, there has been a remarkably increased demand for chemicals of various types. During 1928 the imports of drugs, chemicals, and medicines were valued at £1,969,000, while similar imports for 1927 were only worth £1,141,876. These totals do not include nitrates and various other raw materials imported for special industrial purposes. About £220,000 worth of nitrates for manufacturing are imported annually, while the annual consumption of imported acids is worth about £44,000 a year. Over £400,000 worth of imported sodium is used annually, about £90,000 of sulphur, £35,000 of potassium, and almost the same amount of ammonium compounds. The imports of glycerine for use in manufactures fluctuate between £350,000 and £500,000. The calcium carbide imports are a mere £6,000, as much of this and ammonium compounds being bought by Rhodesia as by South Africa. Various unenumerated imports account for an addition of £600,000.

### Lack of Advertising

A large portion of the South African trade in druggist's supplies and chemicals for manufacturing is in British hands, but it is to be noted with regret that the £904,200 average of the 1920-24 period has not since been reached. By 1927 these imports had fallen in value to £665,189, and this is not altogether due to a reaction against British manufacturers as to a lack of forceful advertising and effective representation, a conspicuous feature of foreign trade representatives in the Union. Canada is finding South Africa a good market for sodium cyanide. Her 1920-24 average exports of this to South Africa were valued at £1,266, but in 1927 their value had risen to £77,189.

A very large part of South Africa's glycerine for manufacturing is bought in France, that country in 1927 selling £116,603 worth in this market. Germany, on the other hand, has not succeeded in capturing her pre-war market for sodium cyanide in South Africa, which was then worth about £220,000 a year, but which in 1927 only yielded £1,572. Holland's sales of glycerine in South Africa were worth £43,211 in 1927. The United States is finding South Africa a good market for drugs, chemicals and druggist's ware, her exports here in 1927 being valued at £134,842. Naturally the market for nitrates for manufacturing purposes is largely supplied by Chile, these sales to South Africa being valued at £221,384 in 1927.

### Market Possibilities

Detailed analysis of these figures is not essential. They are a powerful testimony to the market possibilities of South Africa, a sign that the manufacturer who studies the needs of those he would serve is sure of encouraging returns. It might be mentioned that other British colonies in Africa are good markets for chemical products, Tanganyika Territory, for instance, spending about £50,000 a year in this way.

Of the special South African markets demanding particular attention the Rand Mines is the most prominent. Here, in 1927, £145,158 of chemicals, assay and smelting apparatus was sold, these figures being a decrease of over £5,000 on the previous year's trade. About £304,000 of cyanide is bought annually, and about £16,000 of caustic soda.

The manufacturer must realise that he will find a strong competitor in the manufactured chemical products market in South Africa itself, whose manufactures here now have an annual value of £6,500,000. The total number of factories producing these and allied manufactures is now over 140, the land and buildings used being valued at £1,610,628 and the machinery and plant at £1,788,459. This industry uses annually £3,598,302 of materials, of which £1,069,595 is derived from South African sources. These are the 1927 totals, but the ratio remains much the same. South Africa has long exported drugs and chemicals on her own account, the value of this trade in 1927 being £11,593.

### Duty-Free Chemicals

Although South Africa imposes a Customs duty on many chemicals for the local market, a large number are admitted free of all charges. Among those admitted free are boric and oxalic acids in bulk, alum in bulk and aluminium sulphate, the latter, however, being liable to a suspended *ad valorem*

duty of 20 per cent.; borax in bulk, bromide, annatto, crude glycerine in bulk, manganese dioxide, nitrates (except nitrate of ammonium) for manufacturing or for fertilisers, in bulk, peptone and agar-agar for making bacterial culture media, chloride of platinum, radium compounds, and cyanide of sodium, which is liable to a suspended duty of 8s. 4d. per 100 lb. The duty-free entry also applies in the case of substances for the prevention or destruction of agricultural pests, including sheep and cattle dips and dipping powders, and materials suitable only for dip; substances for the prevention or cure of diseases in plants or trees; sulphate of copper, arsenic, arsenite of soda, arsenate of soda, arsenic pentoxide, carbon bisulphide, and arsenate of lead. Substances for the prevention of wood rot, and substances for the preservation of wood, unless otherwise provided for, and not including stains and oils, are also admitted free of all duty, which also applies in the case of bulk sulphur and sulphurous anhydride. Several classes of chemicals are admitted free, but at the same time are subject to a 3 per cent. maximum duty, which has occasionally to be applied.

### Accident and Disease Statistics

FATAL accidents in the chemical industry reported during November numbered three. In the same month there were reported two cases of aniline poisoning, one case of carbon bisulphide poisoning, and 16 cases of chrome ulceration (two in the dyeing and finishing trade).

## Commercial Intelligence

*The following are taken from printed reports, but we cannot be responsible for any errors that may occur.*

### Receivership

DUROGLOSS POLISHES, LTD. S. Cole, of Sardinia House, Kingsway, W.C.2, ceased to act as receiver and/or manager on December 5, 1929.

### London Gazette, &c.

#### Companies Winding Up Voluntarily

BANK BOTTOM DYEING CO., LTD. (C.W.U.V., 28/12/29.) By reason of its liabilities, December 16. C. P. Spencer, chartered accountant, Palatine Chambers, Halifax, appointed as liquidator.

NORTH WESTERN OIL REFINING CO., LTD. (C.W.U.V., 28/12/29.) By special resolution, December 16. F. Lloyd Williams, 11-13, Victoria Street, Liverpool, chartered accountant, appointed as liquidator.

### New Companies Registered

NON-FERROUS METAL PRODUCTS, LTD., 95, Gresham Street, London, E.C.2.—Registered as a "private" company on December 18. Nominal capital, £10,000 in £1 shares. To promote the production and use of zinc, copper, lead and other non-ferrous metals or their compounds and alloys, and in particular zinc oxides, zinc sulphides, lithopones, zinc alloys, lead, lead pigments, titanium and composite pigments; to carry on the business of manufacturing chemists, manufacturers of and dealers in sulphuric and other acids, alkalis and chemicals; to carry on research work, etc.

THE KLOREX SYNDICATE, LTD., 18, St. Swithin's Lane, London, E.C.4.—Registered as a "public" company on December 19. Nominal capital, £25,000 in 5s. shares. To adopt an agreement with Mexco, Ltd., and the liquidator thereof, and Selection Trust, Ltd., to turn to account the property and rights referred to therein, and to carry on the business of manufacturing and operating chemists, manufacturers of and dealers in salts, acids, alkalis, drugs, medicines, chemical materials and scientific instruments, manufacturers of explosives, gunpowder, nitro-glycerine, dynamite, etc. Directors, A. S. Clift, F. L. Gibbs, E. W. Janson, G. R. Nicolaus.





# Birmingham Supplement

## Chemical Industry in Birmingham & District A Developing Field of Activity

THE chemical industry, together with that of chemical engineering, in Birmingham and district—which includes, of course, the Black Country—occupies an important place in manufacturing activities in mid-England. For a long time the production of various chemicals has been carried on extensively in and around Birmingham, but in recent years there have been big developments, particularly in the domain of metallurgical chemistry. This is due to the multiplicity and variety of the smaller metallurgical industries.

Copper is there smelted, refined, cast and rolled. There is an important nickel and German silver industry; lead and zinc are refined, cast, and rolled; and there is also a large and growing trade in aluminium castings and alloys. The production of non-ferrous alloys is receiving much more attention than formerly. Nickel alloys for electrical and general engineering purposes are featured; and at the present time, in addition to the refining of nickel and cobalt, there is carried on, at one large works, the regular production of rolled sheets, drawn wires, etc., in thirty different nickel alloys.

The demands of the metal-working and other industries have given rise to a lacquer, enamel and varnish industry of considerable magnitude; and much progress is to be noted in the manufacture of cellulose products. In South Staffordshire, paints and colours are made on big scale lines.

### Heavy Chemicals

Most of the heavy chemical industries are situated outside Birmingham; several of importance are at Oldbury and district. At one large works there the manufacture of phosphorus has been successfully carried on for many years; and to-day the range of output is very comprehensive, including phosphorous compounds, carbon tetrachloride (pure and technical), ammonium persulphate, pure precipitated sulphur, calcium phosphide, various brands of aerating materials, together with an important medium for stone preservation and restoration. At another works the manufacture of alkali is carried on, and there is also a large production of ammonia and its salts; of potassium cyanide, which is largely used in the plating industries; and of nitric, sulphuric and hydrochloric acids, which are extensively employed in connection with the galvanising

of iron and the pickling of brass, copper and silver goods in preparation for subsequent treatment. The demand for soldering and welding materials has led to the production of sal-ammoniac, borax and ferrocyanide.

Nor should mention be omitted of the various by-products prepared from the manufacture of coal gas by the Gas Department of the Municipality of Birmingham, whose works are the largest in the provinces. The coke oven plant is distinctive, in that it is the only plant of its kind in the country producing gas which is utilised solely for town purposes. A benzole plant is capable of producing approximately 1,000 gallons of crude benzole a day; and a tar dehydrating plant has a productive daily capacity of 20 tons of refined tar. There are several firms which distil tar.

### Residual Tar Products

Some large firms (including one engaged in iron and steel production) are giving considerable prominence to residual distillation products such as ammoniacal liquor, crude naphtha and benzole, spent oxide, pitch, and crude and pressed naphthalene. From the local manufacture of Mond gas a variety of antiseptic goods are now being successfully marketed; and the many other products of the Birmingham district include oxygen, carbon dioxide, and ferrous oxide; synthetic resins and a variety of moulding materials which have a wide industrial application, especially in electrical engineering; explosives and ammunition; and fertilisers.

### Chemical Plant

The requirements of the chemical industry, in the way of plant, are admirably catered for in Birmingham and South Staffordshire. There are featured, among many other things, petrol systems, stainless and heat-resisting steels, acid-resisting vessels, heat-resisting case-hardening pots, and heat-treatment baths. Chemical laboratory equipment is also made.

The educational facilities afforded at the Birmingham Technical College and the University, in relation to the chemical and allied industries, are excellent. They provide for the needs of all classes of students. The University departments of chemistry, oil engineering and refining, and mining all provide degree and diploma courses; and each maintains a great interest in research.

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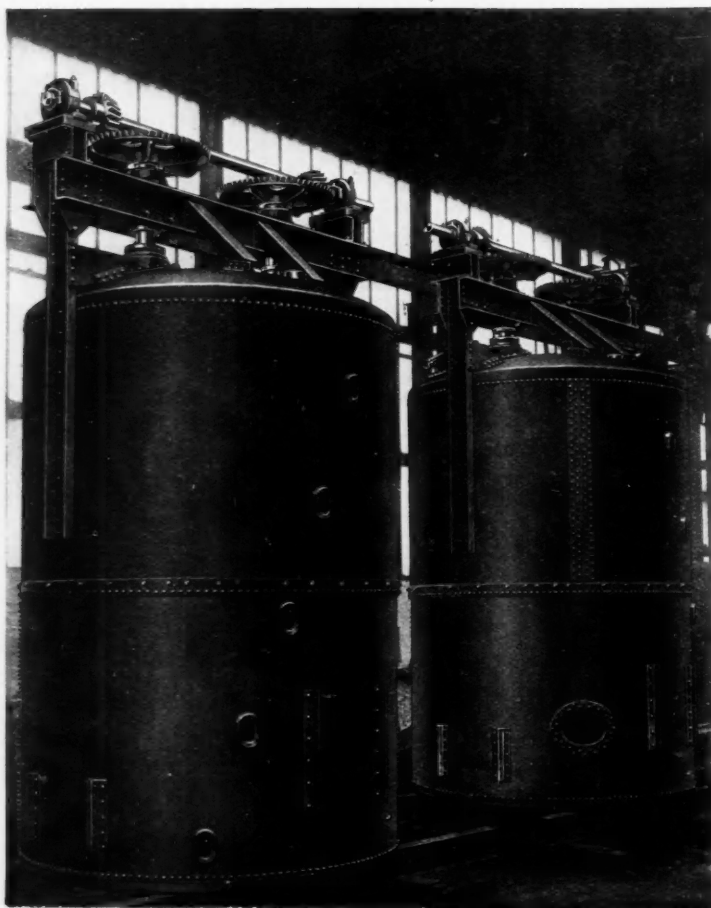
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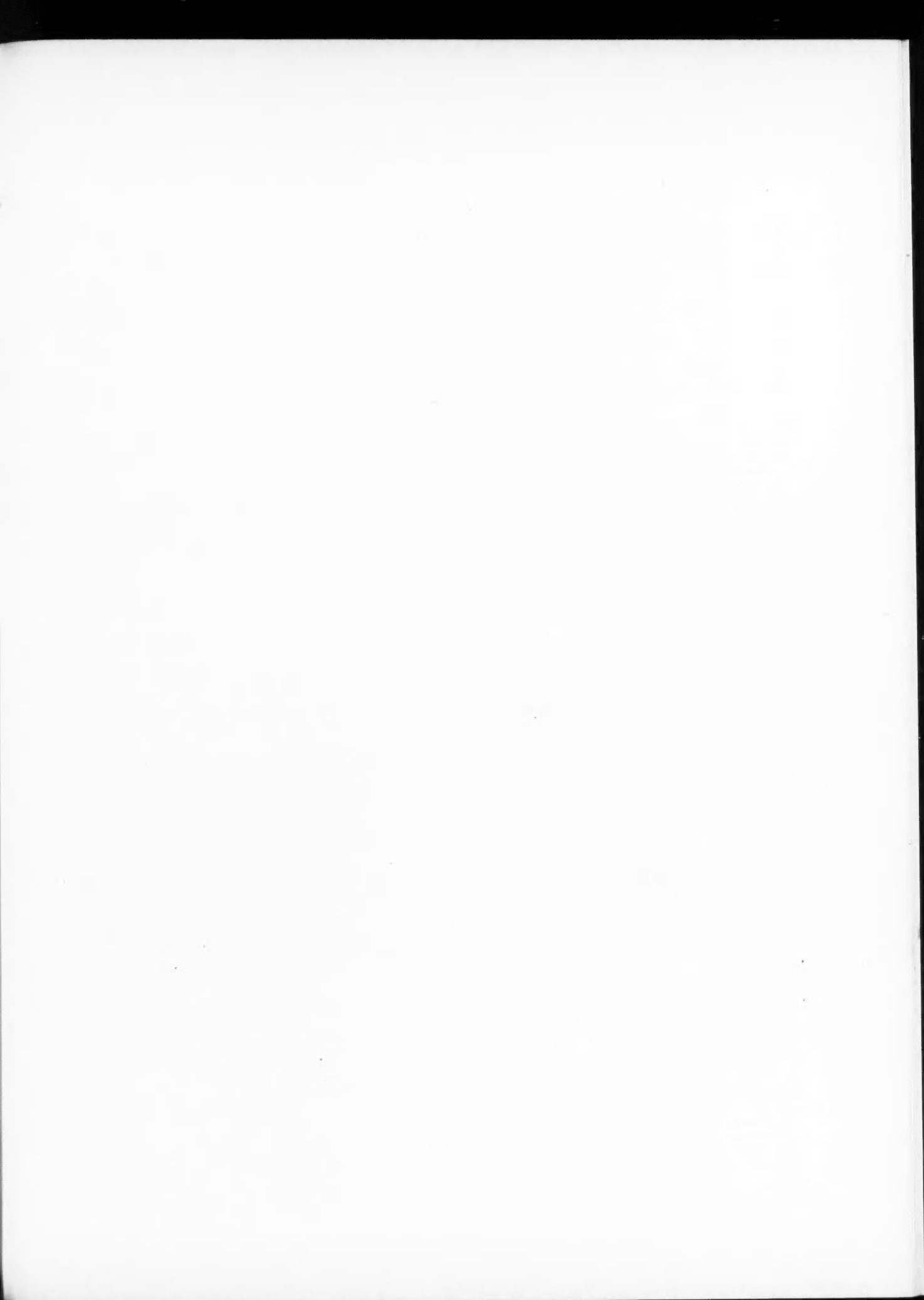
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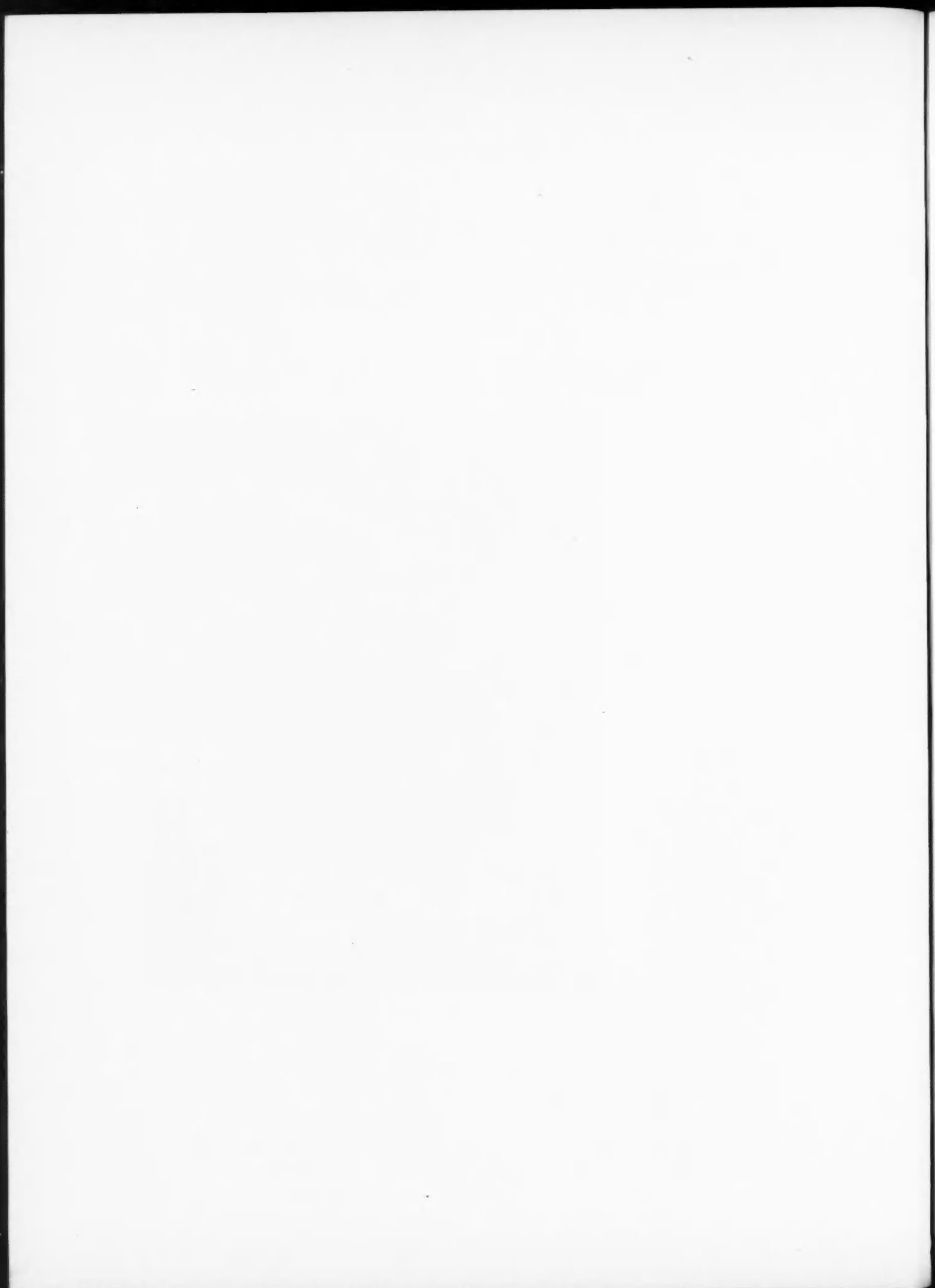
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## Plant and Chemicals: Some Birmingham Products

*A great variety of chemicals, plant, engineering products and kindred materials is manufactured in Birmingham, some idea of which is conveyed by the descriptive notes appearing below.*

### Chemical Plant and Engineering

IN pre-war days, Windmill Engineering Works, Dudley, the headquarters of John Thompson (Dudley), Ltd., was chiefly noted for its self-supporting chimneys. These chimneys were, and are, being made in a variety of sizes up to 13 ft. diameter, and 240 ft. height, plain and ornamental. Some have provision for three or four separate flues at the base, and in special cases often at higher altitudes also. It is worth remarking here that during the prevalence of the recent gales, which are said to be the worst experienced since meteorological records have been kept, not one out of almost a thousand of this firm's chimneys has been reported damaged, although some have been standing over twenty years.

As one goes through the works, however, the outstanding features seem to be the special provision for manufacture of all kinds of chemical plant, homogeneous lead lining, and the lavish use of welding apparatus. The variety of work is great, vessels of all shapes, cylindrical, square, rectangular, hexagonal and various combinations being made, as well as pipe mains, bends, launders of intricate shapes, gas scrubbers, heat exchangers, condensers, separators and gas coolers; while attention may also be drawn to the production of grinding mills, evaporators, filters, drying machines and acetone tanks.

Of interest to paint and colour makers are gumpots, varnish tanks, and various kinds of jacketed vessels, while the general engineer is catered for as regards vertical boilers, muffle furnaces, elevators, hoppers, bunkers, buoys, siren turrets, dust arresters and even lightning conductors.

The firm also supplies oil plant, including petrol tanks, stills, fractionating towers, soda bubbleers, oil storage and carriage tanks.

Among the many different types of acid-resisting plant manufactured there may be mentioned stainless steel tanks, rubber-lined vessels, cast-iron autoclaves with special enamel, and zinc-coated plant; while further important members of this group are homogeneously lead-lined vessels and pipes. The lead-lining department is separately housed, incorporating a sand-blasting plant and chamber to take the largest size vessels, also special apparatus producing pipes homogeneously covered with lead either inside, outside or both. Much care and attention is given to the question of acid-resisting material in the works, laboratory and designing departments.

The firm has a special chemical plant office. This department exists solely for the purpose of designing and developing plant of either standard or special construction to client's requirements.

### Steel and Other Tubular Products

THE Coombs Wood Tube Works, Halesowen, which began operations in 1859, is the principal English works of Stewarts and Lloyds, Ltd., and covers some 40 acres. It has a weekly output of some 3,000 tons of steel and other tubes from  $\frac{1}{4}$  in. to 42 in. bore, in addition to large quantities of special fittings. The range of products cover practically all the requirements of commercial users of tubular goods, and is completed by the other works of the company, with which close collaboration is maintained, and from which large quantities of tubes are obtained to be worked up into a more highly finished form.

Although almost every kind of tubular product is used by chemical manufacturers, naturally there are some which are of more interest than others. Stewarts and Lloyds, Ltd., have always been known for their coils, of which they are prepared to make every known shape and type, however intricate. The limits to the size of coils that can be supplied without joints are imposed not by the limitations of the company's ability to manufacture, but by the lack of facilities available by road and rail for the transport of the finished coil. Special welding work has always been a feature of the Coombs Wood Works, where was installed one of the first electric welding plants in this country. At the present time fire, electric, and oxy-acetylene welding are all employed, the choice of the process being governed by the nature of the work, and special

welding fittings of all types, for all practicable working conditions, are regularly manufactured.

Main steam pipe installations are yearly becoming of more interest to chemical engineers, as the demands of chemical processes for power steadily increase. Coombs Wood Works recently supplied flanged steam pipes for the most severe working conditions yet achieved in this country—815 lb. per square inch pressure at a steam temperature of 830° F. Other manufactures that may be specially mentioned are cylinders for liquifiable and non-liquifiable gases, ammonia tubes, hydraulic tubes; and for the conveyance of gas, air, water, oil and steam, tubes with an immense variety of joints. Summing up, it may be stated that the works produces everything of a tubular nature.

### Blowers and Boosters

ALLDAYS AND ONIONS, LTD., have been manufacturing Roots type blowers and boosters for upwards of 30-40 years, but some five years ago they purchased the goodwill, patterns, etc., of the business of Thwaites Bros., of Bradford, who were also manufacturers of this class of blowing apparatus, since when the business has increased tremendously. The range of blowers manufactured is very varied, consisting as it does of high-speed machines mounted throughout on roller bearings, suitable for pressures up to 5 lb. per square inch, similar machines suitable for a maximum pressure of  $1\frac{1}{2}$  lbs. per square inch, low speed machines suitable for 5 lb. pressure, and low-speed machines suitable for  $1\frac{1}{2}$  lb. pressure. In the case of the low-speed high-pressure machines, these are ring oiling bearings, and cone-bearings in the case of the low-pressure machines. When the blowers are used for boosting gas or handling of any description, they are fitted with bearings remote from the blower, the endplates being gland packed. The Roots blower has been applied successfully for the purpose of cupola blowing, smithy blast, pneumatic transmission, and pneumatic conveying, drying and solvent recovery plants.

In addition to the Roots type blowers, the firm specialise in the manufacture of fans for all purposes, and a competent staff of technical engineers is available to deal with all inquiries that call for special designs. Other of the firm's productions include furnaces, pneumatic and steam hammers, and complete foundry and smithy plants.

### Heat-Resisting Laboratory Glassware

A SUBSTANTIAL amount of factory space and time is given up to the manufacture of laboratory beakers and flasks by Chance Brothers and Co., Ltd., of Smethwick, near Birmingham. These beakers and flasks have to be made of a "metal" (as glass in bulk is termed) that is expensive to mix and has to be melted in a furnace of extraordinary heat. This heat is so fierce that while the glass is being picked up on the blow-tube the glass blower has to wear a mask over his face to protect his face and eyes from being scorched.

Chance's beakers and flasks are blown. Moulding tends to make the glass far too thick, and with a rough surface that collects chemicals and is difficult to clean. It is quite a common thing in a laboratory to see heavy beakers and flasks with a yellow discolouration, and so encrusted with chemicals that they would take many hours to clean. Thin glassware expands steadily and evenly so that sudden extremes of temperature do not put their strain on certain isolated parts. Another factor that has to be carefully watched in the manufacture of laboratory glassware is the "annealing" or cooling of the vessels. Some kinds of optical lenses are bricked up in annealing ovens and left for several weeks to cool in order to achieve the necessary clarity, hardness, and freedom from blebs or air bubbles. Resistance glass does not need such slow annealing as this, but it is nevertheless a long and anxious process, as the slightest hitch can ruin many hundreds of flasks. Notable evidence of the care with which this laboratory glassware is made is afforded by the National Physical Laboratory report. In three separate places the glass is described as "very satisfactory"—high praise from a body like the N.P.L.

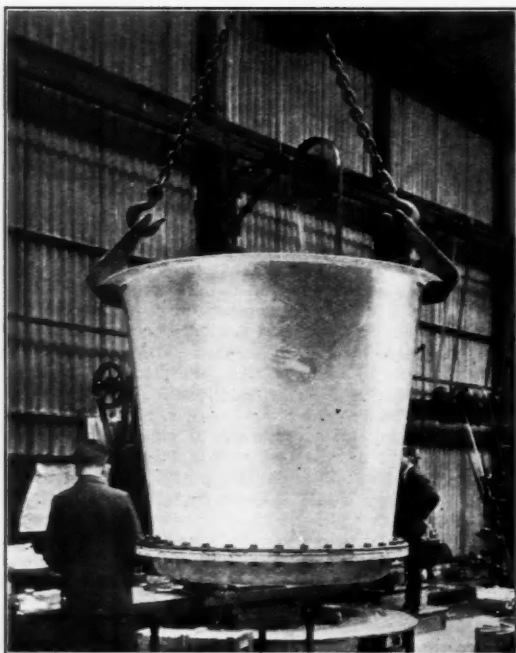
Chance's are almost the oldest glass-making firm in this country. All the glass in the Crystal Palace was made by them. They built the Eddystone Lighthouse and supplied the lenses and reflectors. Indeed, there is probably not a lighthouse round our coast in which Chance's glass is not employed. The latest development of the firm is the introduction to the public of Orlok oven-table glass—cooking dishes made of very similar "metal" to that of the beakers and flasks.

### Resistant Seamless Tubes

THERE is to-day a growing demand for tubes made from materials possessing great strength at high temperatures and having corrosion-resisting qualities when in contact with most acids in solution. The Talbot-Stead Tube Co., Ltd., of Birchills, Wallsall, have for some years devoted themselves assiduously to the manufacture of seamless tubes having such properties. Apart from their extensive business in ordinary mild steel tubes they are now producing tubes made from five different kinds of metals to suit varying conditions and requirements, some of which are in the chrome-nickel low carbon steel class and some in the nickel-copper alloy class. 18/8 steel, containing 18 per cent. of chromium and 8 per cent. of nickel, possesses a great degree of corrosion-resistance and great resistance to scaling at high temperatures. Another class of chrome-nickel steel is that known as 18/8/1, containing 18 per cent. chromium, 8 per cent. nickel and 1 per cent. tungsten. The addition of tungsten adds considerably to the heat resisting qualities of this material, which possesses all the qualities of the 18/8 class with the addition of greater heat resisting power. Tubes made from the 18/8 class of steel, while possessing great powers of resistance to most corroding media, are particularly good where nitric acid is the agent, and for this particular purpose the Talbot-Stead Tube Co. have made large quantities for the chemical industries.

### Aluminium Plant

THE manufacture of plant and equipment, under the broad heading of aluminium, for chemical, foodstuffs and varnish manufacturing and many other industries, is represented by



VARNISH POT OF SHEET ALUMINIUM, WITH SPECIAL BIRMALITE ALLOY BOTTOM PIECE FOR DIRECT FIRING.

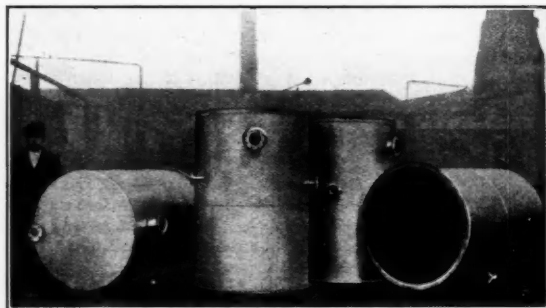
Birmal Chemical Engineers, a branch of the Birmingham Aluminium Casting (1903) Co., Ltd., whose works are situated in Smethwick, near Birmingham. Plant and equipment is produced in welded sheet aluminium and cast aluminium

alloys, and special aluminium alloys have been developed for heat, acid and general corrosion resistance. Complete plants for the refining of edible oils by the continuous distillation process employing aluminium and aluminium alloys throughout are manufactured and installed.

One alloy offered by Birmal Chemical Engineers, known as "Birmalite" Alloy, is used for the construction of vessels for direct flame firing at temperatures of the contents of the vessels as high as 300° C. This alloy will withstand continuous heating over long periods, and after several years of such use shows no sign of damage. Its life may be described as practically unlimited. It is unaffected by free fatty acids, varnishes, gums, etc., and is used for the making of distillation vessels to be fired directly, heat interchangers, varnish pots, etc. "Birmabright" alloy is claimed to resist the corrosive action of hard and salt waters, fruit acids, acetic acid and acetic anhydride, etc. Birmal Chemical Engineers have also an interesting series of "Elektron" alloys. The term "Elektron" covers a range of different magnesium alloys suitable for various constructions, and they are available in the form of castings, extrusions, forgings, etc. In direct contrast with aluminium, "Elektron" alloys are not attacked by alkalis and solutions of caustic alkalis.

### Stainlessly Welded Chemical Plant

MODERN methods of chemical manufacture make heavy demands on plant. It has therefore become of importance to construct plant in stainless steel and non-corrodible alloys, which necessitates welding. Thompson Bros. (Bilston), Ltd., of Bradley, Bilston, specialise in homogeneously welded stainless steel and other non-corrodible alloys. The handling of



GROUP OF 450-GALLON CYLINDRICAL STAYBRITE STEEL TANKS.

such materials requires long experience. The methods of welding used by the firm maintain complete homogeneity, the weld being as stainless as the parent metal, thus providing plant which is consistently non-corrodible over the whole area of its surface. All types of plant are constructed, and the technical department of Thompson Bros. investigates every problem set by inquirers, in order to guide the works in meeting customers' inquiries.

### Fire Extinction

THE manufacture of "Valor" fire extinguishers and accessories forms a very important branch of the activities of the Valor Co., Ltd., of Bromford, Erdington, Birmingham. The following types are produced: The "Valor-Fydrant" chemical fire extinguisher (plunger pattern) for use in garages, workshops, factories and houses; the "Valor-New Era" chemical fire extinguisher (plunger pattern); the "Valor-Fyrou" chemical fire extinguisher (reversible pattern); the "Valor-Foamera" fire extinguisher (foam pattern), using foam as the extinguishing medium; the "Valor C.T.C." fire extinguisher (motor and electrical pattern: this uses a mixture which is a non-conductor of electricity), and also a special power station pattern of "Valor C.T.C." extinguisher (which uses carbon tetrachloride). In addition, the company makes fire brigade hand pumps; fire buckets; and chemical fire engines (foam and soda-acid patterns).

The Valor Co. also manufactures oil storage and distribution equipment, oil filters and settling tanks. This type of plant includes oil cabinets, drums, pumps, tanks, etc.

## Cellulose Enamels and Lacquers

MUCH is heard in these days of cellulose finishes, and it is generally their use on motor car bodies that is discussed. It is not widely realised, however, that cellulose enamels and lacquers have been manufactured for over 40 years by the Fredk. Crane Chemical Co., Ltd., of Bordesley Green, Birmingham. For a time, these finishes were only applied to metals, and "Zapon" enamel became a well-known finish for use in the protection of brass, nickel, oxysilver and similar metalwork. Following improvement in the manufacture of spray apparatus, "Cranco" wood finishes, enamels, and aluminium paint were manufactured and used extensively for finishing metal, wood, glass, leather, composition and paper work. The quick-drying nature of all the "Cranco" products, and their high protective qualities enable most attractive results to be obtained on all types of article.

The call for improved appearance in all classes of work, whether large machinery or small fancy goods, has resulted in the introduction of Crane's cellulose finishes as trouble-free coatings, which retain their attractive appearance. Experimental work is being continually carried out at Bordesley Green, and an efficient service of advice in all matters relating to finishes has been established for the benefit of customers.

## Recording and Measuring Instruments

THE Integra Co., Ltd., of 183, Broad Street, Birmingham, are the sole British agents for the Leeds and Northrup Co., of Philadelphia, who have specialised for years in the manufacture of first-class electrical measuring instruments. Among these instruments may be mentioned potentiometer pyrometers of the indicating, recording and signalling types. These instruments are becoming more and more in favour among progressive firms. Their outstanding advantage is that the readings are absolutely unaffected by any change in the electrical resistance of the thermocouple circuit, and also that the instrument itself can be checked in a few seconds.

Automatic temperature control is also a speciality. Other Leeds and Northrup apparatus worth mentioning are the pH indicators and recorders, conductivity indicators and recorders, resistance thermometers, optical pyrometers, etc. The Integra Co., Ltd., is also marketing in the British Isles the two Leeds and Northrup patented heat treatment furnaces known as the Hump hardening furnace and the Homo tempering furnace.

Among the instruments manufactured by the Integra Co., Ltd., itself, are the gas and air flow recorder, graduated in cubic feet per hour with a 10 in. wide chart, the steam flow indicator and recorder combined, gas and air pressure indicators and recorders, steam pressure recorders, pressure and vacuum gauges, and water flow indicators and recorders combined. All these instruments are built to combine the very often opposing qualities of accuracy and robustness, and are admirably suited for industrial measurements.

## Oils, Paints and Chemical Products

W. J. TRANTER, Ltd., of Tibbington Works, Princes End, Tipton, Staffordshire, established as long ago as 1890, are oil refiners and chemical manufacturers on a very large scale, marketing a very wide range of products for use in all branches of the engineering and allied trades.

One department is for the manufacture and blending of all classes of lubricating oils and greases, cutting compounds, soluble oils, screwing oils, drawing mixtures, hot and cold neck greases, "Titan" brand motor oils and greases. A portion of the works at Tipton is devoted to the manufacture of paints and bituminous solutions of all colours, aluminium and gold paints, and acid-resisting black, which is specially suitable for all kinds of ironwork, acid tanks, etc. Another department deals with non-conducting composition for retaining heat in boilers and steam pipes, etc. The selection of the most suitable composition consistent with efficiency and cost is of great importance, taking into consideration the high cost of fuel and the high temperature of steam. The non-conducting coverings are supplied in plastic and sectional form, and can be finished off with canvas, bitumen sheeting, zinc metal, black mild steel, galvanised steel or planished steel sheeting. Some

of the largest covering contracts placed in this country and abroad have been executed by this firm with entire satisfaction.

All steam users having Lancashire boilers and troubled with scale and mud should carefully consider the installation of Tranter's patent sediment ejector in their boilers. This apparatus is attached to the blow-off inside the boiler, and when in operation ejects the mud and concentrated solutions along the whole length of the boiler. About 100 sets have been installed during the past two years.

W. J. Tranter, Ltd., are contractors to some of the leading firms in the engineering and automobile industries, and carry large stocks of their various products at the Tibbington Works, Tipton, which is excellently situated in regard to transport facilities.

## Enamelling, Drying and Other Stoves

FOR many years, Messrs. Silas Hyde, of Evelyn Road Works, Greet, Birmingham, have made an exhaustive study of stoving methods for all classes of enamelling, lacquering and drying. The design of their double and triple-cased stoves for enamelling, coupled with the registered design atmospheric burners, effects a big saving in time and overhead costs, besides ensuring a uniformity in results obtained. These stoves are made of best quality C.R.C.A. patent flattened steel sheets, and the walls are lagged with high-grade silicate of cotton. They have the advantage of being built in section and can be assembled on site, even by inexperienced workmen. The double-cased stove is strongly recommended for ordinary stove enamelling for the motor, motor cycle, and other trades where first-class finish is essential. The triple-cased stove is recommended for colour enamelling and lacquering and can be adapted for all classes of industrial drying and heating processes, including armature and coil baking for electrical apparatus, drying transfers, food and chemical products, etc.

Special attention is drawn to the Silas Hyde rotary low temperature drying oven. It is designed for the quick drying of articles on a repetition basis. It is cylindrical in form with a rotating inner case containing six segments, each of which has three shelves. The speed is controlled by an electric motor and can be so regulated that one revolution of the cage completes the drying process. Although the loading position is always open the design of the stove is such that there is no loss of heat occasioned thereby. The oven is triple-cased in the rear portion and double-cased at the front. It is highly recommended for all trades where repetition work and a uniform high class of finish is essential.

The company also manufactures tube benders' stoves, blueing and bronzing stoves, track runway stoves, cosletting plants, shaking barrels, cyclone dust and shaving separating plants, and blast work for forges, and undertakes all classes of sheet metal work.

## Metal Sheets, Tubes, Wire, Etc.

AN existence of over 150 years' duration is the remarkable record of Charles Clifford and Son, Ltd., of Fazeley Street Mills, Birmingham. The firm was established in 1776, and is one of the oldest in the non-ferrous metal industry. Copper, brass and yellow metal in the form of tubes, sheets, rods and wire have been for many years its chief products. In recent years a considerable and increasing demand has arisen for bronze for innumerable purposes, both mechanical and ornamental, and in various qualities. Their popularity is due to the fact that bronzes possess high resistance to atmospheric and chemical corrosion, combined with strength approaching that of steel.

Phosphor bronze may be said to be essential to the engineering and chemical industries, and new uses are constantly being found for it beyond the ordinary and general requirements. Besides sheet, strip, and wire, which were among the earlier forms in which this alloy was produced, cold-drawn rods and solid-drawn tubes are now in regular demand, and in all of these forms the firm specialises. High conductivity copper strip and bus bars, which are largely used in the chemical industry, are also produced. The firm issues a list which gives details of the manufactures, as well as much useful information respecting weights, gauges, etc., and a copy will be sent to any reader on request.

### The "Grit Nuisance" Problem

THE emission of grits and dust from chimneys and stacks is a trouble well-known to engineers, and a nuisance to the public who live in a manufacturing district. The apparatus known as the patent "Centriflovan" grit catcher, manufactured by the Tipton Tub and Tube Co., Ltd., of Tipton, Staffs, is designed to cope with this difficult problem. Hundreds of these catchers have been installed on the stacks at gas and electricity works, preventing the roofs from being covered, and gutters and drains from being stopped up, also relieving the public of the nuisance of the grits being blown into their homes. The apparatus is extremely simple in design and quite automatic in action. The grit-laden gases ascend the chimney to the catcher, and by the construction of the interior of the catcher the gases are given a rotary and downward motion. Advantage is therefore taken of centrifugal force and gravity to separate the grits, which are deposited in an inner cone in the catcher, whence they are conveyed by a pipe to a grit receiver at the base of the stack. The quality of the grits varies according to the type of plant, method of firing, and fuel used, but the amount of fine grit which passes through a 200 mesh shows the efficacy of the patent "Centriflovan." Many thousands of tons of grit are being caught annually.

### Power Plant

IN many branches of the chemical industry large quantities of steam are required for process work. These are favourable circumstances for the production of very cheap power, as the steam can first be passed through an engine or turbine to provide power before being exhausted to the process main. Power is thus virtually a by-product, and is obtained for the same expenditure of steam as is required in any event for the process. Belliss and Morcom, Ltd., of Ledsam Street Works, Birmingham, have specialised in power plant for these conditions, and have supplied upwards of 1,450 engines and turbines of the back pressure or pass-out type for operating on this system alone. Belliss and Morcom engines are of the totally enclosed forced lubricated type, as originated by the firm in 1890. They are very simple in construction and will operate for years with a minimum of wear. Cases are on record where large Belliss engines have operated for as long as three years absolutely non-stop. The full list of the company's manufactures includes steam engines, steam turbines, condensing plants, air and gas compressors, vacuum pumps, Diesel oil engines, paraffin oil engines, and pneumatic hose couplings.

### Motor Vehicles

THE AUSTIN MOTOR CO., LTD., of Longbridge Works, Birmingham, in its programme for the coming year, is adhering to its policy of placing efficiency before fashion. The company considers that it would be a retrogressive step to make improvement an annual event. It prefers to keep its cars right up to the minute, and is sufficiently considerate of its already very wide public to make practically every improvement in such fashion that it may be incorporated at reasonable cost in old cars, and thus enable the owners to keep abreast of the times. Visitors to the recent Motor Show noted that there were a number of alterations and improvements which had been incorporated since the last exhibit. External metal fittings on all cars are now chromium plated. Windscreens of Triplex unsplinterable glass have previously been a standard fitting on all Austin cars. Saloon cars now have all windows Triplex fitted in addition to the screens of all models.

The Twelve and Sixteen models are fitted with a new ball type gear change. The gas and ignition controls are now placed neatly at the head of the steering column on the Twelve, Sixteen and Twenty models. The smart Bakelite fitting replaces the quadrant and is not only a useful but a pleasing innovation. The new biflex headlamps of the Sixteen and Twenty models are fitted with dip and switch-reflector equipment electrically controlled.

The pressed steel framework affords the maximum strength with the minimum weight, and its use has also enabled the production of bodies on closed models that permit unobstructed vision of the road. This is a most important factor in the maintenance of safety first. Aluminium is used to an even

greater extent than heretofore, which means further saving in weight. Special attention has been given to the important factor of floor covering. No fumes are able to enter the closed body and there is an entire absence of draught. Deflector ventilators are fitted in the scuttles of all models. The colour schemes are in keeping with the most modern requirements and yet carry Austin distinctiveness—marked by the best of taste. There is a generous choice of colours and colour combinations.

Among the products of the company are the 20 h.p. six-cylinder "Ranelagh" limousine; the 16 h.p. six-cylinder fabric saloon (sunshine roof); the 16 h.p. six-cylinder Burnham saloon; the 12 and 7 h.p. tourers; and the 7 h.p. coachbuilt saloon.

### New Smoke-Preventing Baffle Bridge

IN connection with the operation of "Lancashire" and other industrial boilers and the prevention of black smoke, it is well known that most designs of hollow baffle bridges, generally of firebrick, with secondary air admission, have been troubled with dust deposits and insufficient mixing of the secondary air with the unburnt products.

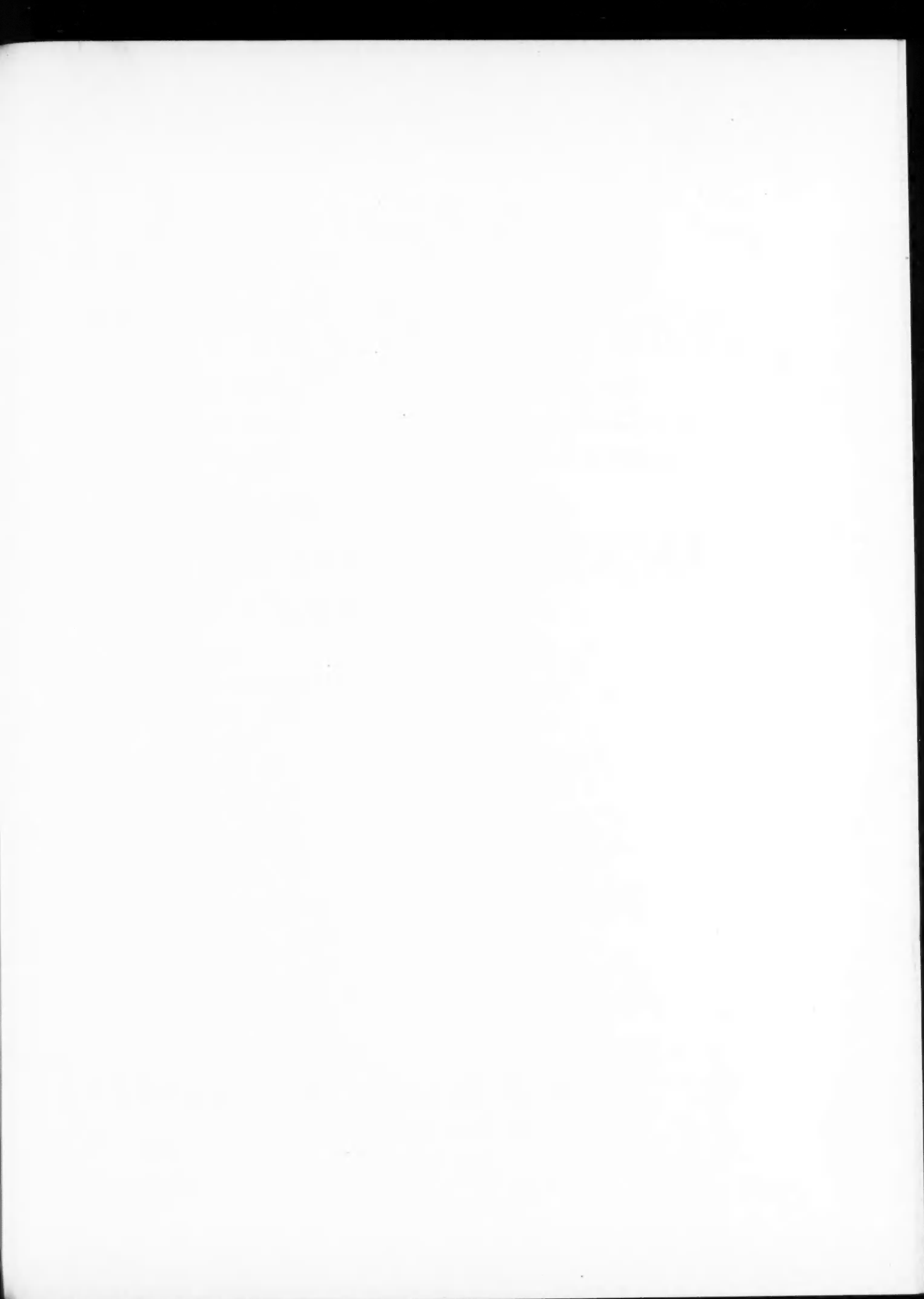
It is interesting, therefore, to know that the "Turbine" Furnace Co., Ltd., have now devised a new and ingenious hollow perforated baffle bridge, first shown at the recent Shipping and Engineering Exhibition, Olympia, made of interchangeable cast iron sections instead of firebrick, fixed side by side, in which it is impossible for dust to lodge and cause trouble. Also a considerable part of the air for combustion mixed with steam from the nozzles passes through the bridge under "blast" conditions, and as a result the life of the latter is almost indefinite, like the firebars themselves, because of the cooling effect of the air and steam.

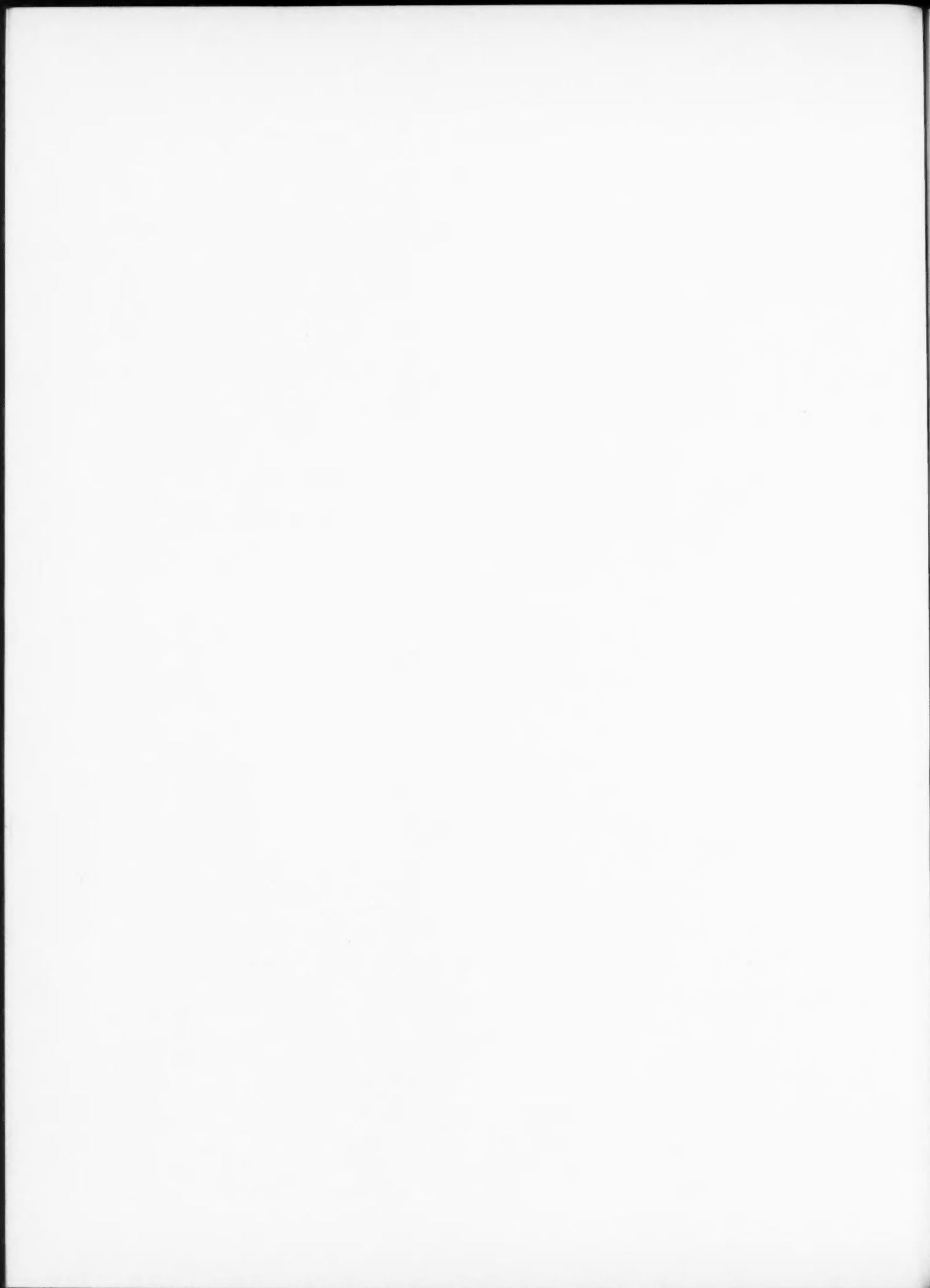
The bridge is constructed of a series of cast iron sections, each triangular shaped in side view, 2 in. wide on the face and weighing about 27 lb., forming a sloping bridge or grate surface resting on the last four rows of transverse firebars in the standard "Turbine" furnace. For a normal width, such as that used in "Lancashire" boilers, say, 3 ft., the number of sections comprising the bridge is sixteen, along with two end pieces, giving fifteen rows of slots side by side, six in a row; a total of ninety slots. In normal operation, the bottom two rows of these slots are covered by the fire, leaving above four rows, or sixty slots free for the passage of the steam and air blast. This passes in from the main trough bars by way of the spaces between the last four sections of the bars and then out to the fire, burning all the smoke and unconsumed gases on the usual "secondary air" principle. Another advantage claimed is that the latter air is thoroughly diffused because of the sixty slots or so, thus giving immediately an effective mixture with the combustion products, while also, unlike many secondary air devices, there is a considerable amount of steam present which aids combustion and prevents the adherence of clinker.

As regards ash and dust, such material only passes to a limited degree through the slots because of the shape and angle of inclination, while at the bottom of the bridge is a damper device operated by a rod from the front of the boiler. This allows any accumulated dust to fall out into the boiler furnace tube, while there is included a second damper device, so that in the case of abnormally smoky coal an extra volume of cold air can be passed through the baffle bridge, although this is rarely necessary. No steam jets are necessary at the back of the bridge, while the new arrangement is easily added to the existing "Turbine" furnace installation.

### Textile Chemicals

STOCKS of numerous textile chemicals and allied products are kept by M. G. White and Co., of Canal Road, Bradford. This firm are direct importers of formic acid, sodium perborate and tannic acid. In addition to handling all classes of dyestuffs, they are importers of many specialities such as Hydrophthal and Neomerpins, which are among the latest scouring, wetting-out and penetrating agents used in the textile industry: also Egalisal and Egalisal K., which are claimed to give protection to wool against the action of all chemicals in scouring, dyeing, garment dyeing, and the shoddy industry.





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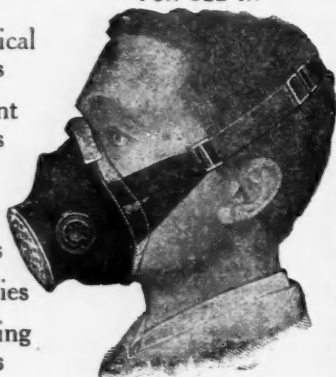
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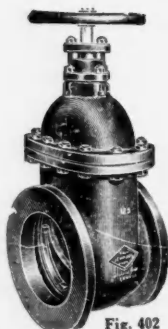
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## Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

*Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.*

### **British Synthetics and Naphthol Products**

OUR attention has been drawn to the possibility of certain statements published in our last *Dyestuffs Monthly Supplement* relating to British Synthetics and Naphthol products being misinterpreted or misunderstood. In order to prevent any such possibility we have pleasure in publishing the following statement, which, we are assured, gives the facts of the position as regards British Synthetics, Ltd. :—

"British Synthetics, since its inception in 1923, has manufactured products of the Naphthol AS class under the trade name of Brenthols under its own patents and has never manufactured under licence. The agreement with the I.G. was made on account of the Application Patents of the I.G. and is a reciprocal agreement, and does not place British Synthetics solely in the position of merchants, as might possibly be assumed from your correspondent's remarks. British Synthetics are now manufacturing on a very substantial scale, and had it not been for their patents and their entry into this field of dyestuffs it is to be presumed that there might have been no British manufacture of products of the Naphthol AS class to-day."

### **Sir Henry Sutcliffe Smith's Address**

The addresses given by Sir Henry Sutcliffe Smith in his capacity as chairman of the Colour Users' Association have always commanded great attention, and deservedly so, for they are invariably clear and reasonable reviews of the dyestuff situation. This year's address, delivered at the annual meeting of the Association, was the tenth delivered by Sir Henry.

The price of dyestuffs is a subject of unending interest alike to the maker and the consumer. Never before, however, has Sir Henry, in his frequent references to the matter, been able to say, as he did at the Association's annual meeting, that the prices of dyestuffs in this country were not substantially higher than those in force on the Continent, and that, in fact, prices in some Continental countries were much higher. To this we may add the view of our Lancashire market correspondent, that while prices are still falling in some cases, they have not yet become stabilised.

### **The Dyestuffs Act**

The most important part of Sir Henry's address was that dealing with the approaching expiry of the Dyestuffs (Import Regulation) Act of 1920. This Act will expire in January of 1931, and statements made recently by those interested in the matter have been marked by considerable caution. Sir Henry points out that consumers have, through the past eight years, made a considerable contribution towards the establishment on a solid basis of the British dyestuff manufacturing industry, but that the industry is now rapidly approaching the point at which it can face world competition. He supports this view with the statement of Lord Melchett, that costs of production in the British chemical industry are at least as low as those in other countries.

On the other hand, Sir Henry expresses the personal opinion that "it would be economic folly to allow this great industry—so necessary for our trade security and now well on the way to be thoroughly established—to decline. . . . It would be a serious blow to our prestige

and to our overseas trade if anything were to arise now which might counteract the efforts of the past eight years." This expression of opinion is a remarkable testimony to the broadmindedness and public spirit of the man who uttered it, and an indication of the new spirit which prevails in industry—a spirit which prompts all who are interested in commodities, whether as manufacturers or users, to think not merely of their own advantage but of the interests of the community at large.

### **The Trend of Events**

It is impossible, at this stage, to offer any forecast of the manner in which the situation will develop. Sir Henry's statement of the case is really a reasonable review of the whole situation as affecting all parties. He renews the suggestion made a year ago that consumers and users should meet in a round table conference. The Dyestuff Industry Development Committee is engaged in the preparation of a comprehensive review of the operation of the Dyestuffs Act since its initiation.

The deciding factor in the situation will, of course, be the attitude of the Government. At the moment, the nature of that attitude is unknown. It was plainly indicated in the King's Speech at the opening of Parliament that the Government does not approve of purely safeguarding duties, and this has been emphasised in various Ministerial replies to questions. For example, Mr. W. Graham, President of the Board of Trade, stated that "with regard to ordinary safeguarding duties, they on that side of the House had always opposed them as a party." It has, however, been suggested in some quarters that some distinction may be drawn between key industry duties and "purely safeguarding" duties. It is now clearly understood by the country as a whole that the chemical industry stands in a very special position. Moreover, its organisation, and the quality of the brain-power at its command, put it in a class by itself. It is quite certain, therefore, that before any action is taken all the facts of the case will be clearly presented to the Government. Nothing more can be said at the moment.

### **The Price of Benzol**

As a result of the imposition last year by the Government of a duty on imported hydrocarbon oils, a very peculiar situation has arisen. The reasons for the duty were, among others, to encourage the home production of oils suitable for use as motor fuels, by new methods of carbonisation and so forth. The benzol manufacturers of this country, in view of the use of benzol as motor spirit, raised the price of their product so as to bring it into line with the increased price of petrol due to the import tax of fourpence per gallon. Benzol and toluol are very important items in the cost of production of numerous chemical products, including dyestuffs, and this increase in price has obviously had very serious effects. So far from encouraging home production, the effect in the dyestuff-manufacturing and kindred industries has been the direct reverse. Sir Henry Sutcliffe Smith, in the course of his address to the Colour Users' Association, appealed to the benzol manufacturers to consider the serious effect of the high cost of benzol and toluol in this respect.

### A Text-Book on Rayon

Sir Isaac Pitman and Sons have just published a second edition, revised and enlarged, of *Artificial Silk (Rayon): Its Manufacture and Uses*, by Thomas Woodhouse (pp. 245, 7s. 6d.). It aims at giving a concise account of the chief operations involved in the separation of cellulose from the raw materials, the making of yarns, and the subsequent processes of winding, warping, sizing, beaming, weaving and knitting. As compared with the first edition the size of the book is nearly doubled.

The chapter headings are as follows: Raw materials and their treatment; commercial varieties of artificial silk and their constituents; general principles involved in the making of artificial silk threads; reeling and drying the threads; the utilisation of artificial silk yarn for hand-knitted and machine-knitted articles; hand knitting, bottle bobbin winding, and cone winding; machine knitting; warping and beaming; sizing, drying and beaming; drawing-in, reeding, gaiting and weft winding; weaving. A feature of the book is the large number of diagrams and photographs of plant used in the industry.

### Claim Before Mixed Arbitral Tribunal

The Anglo-German Mixed Arbitral Tribunal (Division A), consisting of Dr. Helge Kjalstad (President), Mr. Heber Hart, K.C. (British member), and Dr. Robert Wendrin (German member), sitting in London on Monday, delivered judgment, by consent of the parties, for the Bradford Dyes' Association, Ltd., against the German Government for the sum of £29,392 as "cash assets" under Article 297 (h) of the Treaty of Versailles, together with interest at 5 per cent. per annum—as compensation for loss of use under Article 297 (e)—on £17,471 from September 30, 1917, until crediting, and for a further sum of £3,500, as compensation for loss of use under Article 297 (e). Mr. Schiller, K.C., and Mr. D. B. Somervell, K.C., appeared for the claimants; Dr. Huecking (German Government Agent) for the German Government; and Mr. B. Honour (British Government Agent) for the British Clearing Office.

The claimants were the owners of all the shares in S. H. Sharp and Sons, G.m.b.H., of Kingersheim, Alsace, before the war, and the German Government, in the course of its compulsory administration during hostilities, invested certain funds of the latter company in German war loan. After the war, the claimants caused these bonds to be transferred from Strasbourg to the London County and Westminster Bank in Paris, where they still are deposited, and, about the same time, sold their shares in Sharp and Sons, G.m.b.H., to a French company called Blanchisserie et Teinturerie de Thaon for £110,000, expressly reserving their rights to compensation for damage caused by the sequestration. The original claim before the Tribunal consisted of several items, some of which were dealt with in the Tribunal's interlocutory decision of June 25, 1928, and in the further decision by consent of the parties dated April 22, 1929. The main item outstanding was the claim for £29,392, the sterling equivalent of the M.602,750 war loan investment, and it was a condition of the consent judgment that the war loan in question should be handed over by the claimants to the German Government.

### Dyestuff Licences

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during June, has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee: The total number of applications received during the month was 625, of which 509 were from merchants or importers. To these should be added 8 cases outstanding on May 31, making a total for the month of 633. These were dealt with as follows: Granted, 581 (of which 555 were dealt with

within 7 days of receipt); referred to British makers of similar products, 32 (of which 25 were dealt with within 7 days of receipt); outstanding on June 29, 20. Of the total of 633 applications received, 580, or 92 per cent., were dealt with within 7 days of receipt.

### Third U.S. Light-Fastness Report

The light exposure tests on 1,252 dyed samples undertaken by the Sub-committee on Light Fastness of the American Association of Textile Chemists and Colorists, in co-operation with the Government Bureau of Standards, have produced results that justify a lengthy report. Under the title of "The Fading of Dyed Textiles to Daylight and in the Carbon Arc," this report has been prepared by William D. Appel, of the Bureau, and William H. Cady, of the Association.

Twelve hundred and fifty-two specially prepared dyeings on cotton, wool, silk and weighted silk, representing some three hundred and eighty-one different colouring matters, were exposed to daylight in several different ways and to the light from a glass-enclosed carbon arc. Each sample was exposed for four different periods of time. In general, slight fading occurred in the first period, decided fading in the fourth, and intermediate amounts of fading in the other two.

### Italian Intermediate and Dye Industry

According to the United States Trade Commissioner in Rome, the Italian domestic production of coal tar crudes is still insufficient to cover the needs of the industry, and coal tar crudes are imported to yield the intermediates used by the domestic dye manufacturers. The domestic industry supplies about 90 per cent. of the coal tar intermediates consumed by the domestic dye industry, and production of these substances rose from 800,000 kilos. in 1918 to 5,300,000 kilos. in 1926. During 1927 production declined again to 4,500,000 kilos. The principal intermediate produced in Italy is aniline oil, production of which exceeds 1,000,000 kilos. annually. The excess chlorine derived from the domestic electrolytic caustic soda industry finds utilisation in the preparation of the important intermediate chlorobenzene, with its derivatives dinitrochlorobenzene, dinitrophenol, and paranitrochlorobenzene. Other intermediates which are produced in large quantities are: H acid, the production of which exceeds 350,000 kilos.; betanaphthol, with a production of 250,000 kilos.; benzidine, with a production of 250,000 kilos., and paranitroaniline, with a production of 150,000 kilos. Many other intermediates such as tolidine, alpha-naphthylamine, gamma acid, naphthionic and sulphanilic acid, are also produced.

### Present State of the Italian Industry

Italian production of coal tar dyes rose from 975,000 kilos. in 1918 to approximately 7,000,000 kilos. in 1926. A considerable retrogression, however, was registered in 1927, with a falling off of about 820,000 kilos. as compared with the previous year. About 70 per cent. of Italian production is accounted for by sulphur dyes, but a beginning has been made in the manufacture of certain vat dyes. The Cesano Maderno plant of the Acna group has had its synthetic indigo on the market for two years, and the plant has a capacity of approximately 6,000 kilos. daily (1927 production of synthetic indigo is estimated at 1,800,000 kilos. in the form of 20 per cent. paste). Indanthrene dark blue and indanthrene black are also made now at the Cesano Maderno and Conca Fallata plants of the Acna group. So far, however, the Italian industry has not gone in for the production of anthraquinone dyes.

There are about 15 plants in Italy producing coal tar dyes, two-thirds of which specialise in sulphur dyes. Eight are located in the vicinity of Milan, and others near Turin, Cuneo and Bergamo.

## Sir Henry Sutcliffe Smith on the Dyestuffs Situation

### Annual Meeting of the Colour Users' Association

The tenth annual general meeting of the Colour Users' Association was held at Blackfriars House, Manchester, on Friday, July 5. Sir Henry Sutcliffe Smith, the chairman, in accordance with his usual custom, reviewed the dyestuffs situation from the aspect of the users. The more important points touched on are given below.

IN accordance with the usual practice, said Sir Henry Sutcliffe Smith, the vigilance committee of the Association again applied to the Board of Trade for the renewal of the exemption of oxalic acid from the provisions of the Key Industry Duty, and the Board renewed the exemption for a further period of 12 months to December 31, 1929. It seemed circumlocutory that application for exemption from the Safeguarding Act should require to be made annually for such commodities as oxalic acid which are not made in this country. Would it not be better procedure if the exemption lists were allowed to remain intact throughout the full period of the Act, or until specific articles were removed from the list as a result of the usual inquiry? There were several chemicals of serious importance to the colour-using industry afforded a protection of 33½ per cent. duty under the Safeguarding Act, for which the prices ruling in Great Britain compared unfavourably with those in foreign countries. In one important commodity a British maker was selling on the Continent at Continental rates which were considerably less than those ruling in this country. Whilst for various reasons it would be unwise for this important chemical not to be made in this country, there was a serious responsibility placed upon the domestic producer to sell a protected product at somewhere round the world market price.

#### Applications for Dyestuff Licences

During 1928, 7,900 licence applications had been dealt with by the Dyestuffs Advisory Licensing Committee, and the following was a summary of the quantities and values of the licences granted during the period from the commencement of the Committee in 1921 to the end of 1928.

Year	For importation from Germany		For importation from Switzerland		For importation from other sources		Total	
	lb.	Value £	lb.	Value £	lb.	Value £	lb.	Value £
1921 ..	671,032	197,466	1,706,754	763,299	209,719	82,056	2,677,505	1,042,821
1922 ..	1,325,671	375,675	1,038,235	694,740	270,987	33,404	3,234,893	1,103,819
1923 ..	1,817,571	493,499	1,412,616	459,861	461,253	36,177	3,691,440	989,537
1924 ..	1,805,145	308,226	1,191,931	363,513	39,158	9,204	3,039,234	770,943
1925 ..	2,175,262	334,749	1,157,270	307,754	66,522	9,081	3,399,054	651,584
1926 ..	2,949,858	599,157	1,100,951	333,448	91,778	11,462	4,232,587	944,007
1927 ..	3,044,152	719,938	1,230,815	306,595	115,389	16,480	4,990,350	1,034,013
1928 ..	3,534,935	729,393	1,373,226	335,226	122,350	9,494	5,030,511	1,074,113

The figures since 1925 might be taken as representative of the total imports, as down to the end of 1924 Reparation Colour was imported, for which no licences were required.

The licences granted in 1928 showed an increase of 40,155 lb. or 0.85 per cent. over 1927, and although the figures for the two years 1927 and 1928 showed a large increase over the previous years, yet, having in mind the considerable increase in the production of dyestuffs in the United Kingdom during the same period, he did not consider that the increase in the imports *vis-à-vis* British production was unusual. In 1928 the production of dyestuffs in the United Kingdom showed an increase over 1927 of 11,355,324 lb., or 29 per cent., and in 1927 there was an increase of 30 per cent. over 1926.

Last year he drew attention to the necessity for some modification in the procedure for dealing with the application for licences on price grounds. The reduction of the factor from twice pre-war to 1½ times had lessened the difficulty, but, on a point of principle, he contended that where a British manufacturer introduced an equivalent for certain foreign products, the British user should not be called upon to pay a minimum of 1½ times pre-war, if, prior to the introduction of the British product, the foreign price was less. Representations had been made to the Licensing Committee that the British manufacturer should, in such instances, accept for a minimum period of six months the same price as foreign manufacturers had been selling at during the previous three months.

In the case of general applications on price grounds, he would again urge the Licensing Committee to accept the suggestion which had been previously made, that the quotation from a foreign competitor should be submitted only to the chairman and the neutral members of the committee.

#### Dyestuffs Industry Development Committee's Activities

The suggestion he made last year that the Dyestuffs Development Committee might be usefully employed in the preparation of a comprehensive review of the operation of the Dyestuffs Act since its initiation, with particular reference to the progress made and suggestions for dealing with the situation after the expiry of the Act, had been accepted. Both the makers and the users had been asked to submit their views and recommendations. He would again urge the necessity for either the Development Committee, or preferably the Board of Trade, to issue annually a complete report of the production of dyestuffs in this country, on similar lines to the Census of Dyes issued annually by the United States Tariff Commission. It seemed extraordinary that they had to refer to American publications to obtain recent and authoritative detailed information regarding some of their important industries.

#### Benzol and Toluol

Very important items in the cost of production of dyestuffs were the prices of benzol and toluol. These materials were now nearly three times the pre-war price. Part of this serious increase was due to the imposition by the Government last year of a duty on imported hydrocarbon oils. In sympathy with 4d. per gallon tax imposed on imported petrol, the benzol manufacturers of this country immediately raised the prices of benzol and toluol a proportionate amount, and further additions had been made subsequently. Surely it was not intended by the Government that a hardship should be imposed upon the firms in the dye-making industry which used benzol and toluol as raw materials?

#### Growth of the British Dyestuffs Industry

It was gratifying to observe the growing activity of the dyestuffs industry in Great Britain. From statistics compiled by the Board of Trade issued recently, the total production of dyestuffs by the principal British makers aggregated 50,907,080 lb., being the substantial increase of 11,355,324 lb., or 29 per cent. as compared with 1927.

Included amongst the many new products put on the market by British manufacturers during the last twelve months were some of outstanding importance. Caledon Browns G & R were the equivalents of the fastest and most universally used browns of the vat series—Indanthrene (Algoles) Browns G & R respectively. These two colours had for many years been indispensable for the production of fast shades on vegetable fibres. Of lesser fundamental importance, but still of considerable moment by virtue of their wide application in wool dyeing, were the "6B" brands of Acid Milling Violet, equivalent to various "6BN" brands of Acid Violet of Swiss and German manufacture hitherto in very large consumption, and Solway Blue R, equivalent to the widely-used anthraquinone acid wool colour Cyananthrol R. During 1928 the British production of vat dyestuffs (excluding indigo) was approximately four times the total consumption of such dyestuffs in this country during 1913.

#### Fastness

Developments in the production of coloured fabrics possessing the requisite degree of fastness to all the ordinary conditions of wear and tear were still in their infancy, and a large responsibility was placed upon all sections of the British textile and finishing trades to secure that in these developments this country, with its enormous export trade in textile goods, played its proper part. Co-operation for this purpose was essential, and a representative committee formed by the Society of Dyers and Colourists had for some time past been considering the question.

The use of acetate silk for textile fabrics, both alone and as a mixture with cotton, wool and silk, had introduced into the dyestuffs and dyeing industries problems of an entirely novel nature. It was only right that they should acknowledge the really excellent pioneer work done by the British Dyestuffs

Corporation in the production of suitable dyestuffs for this new fibre.

#### Prices of Dyestuffs

He had dwelt at some length in all his previous addresses on the question of the price of dyestuffs, and there was evidence to shew that, in consequence of the Dyestuffs (Import Regulation) Act, 1920, by which they were fostering the establishment and maintenance of the dyestuffs manufacturing industry in this country, the colour using industries had to pay a price for their commodities much in excess of the world price. That burden has been cheerfully borne as part of their contribution towards the establishment of the dyestuffs industry. It was not to be expected, however, that the British user could continue indefinitely to pay a price substantially in excess of any of his world competitors.

Three years ago, the Association sent a deputation abroad to inquire into the ruling prices of dyestuffs, and as a result of that inquiry it was found that British users were paying prices in excess of their foreign competitors, particularly in the newer and faster types of wares. To bring the information down to date, further inquiries had recently been privately made by two leading firms. The results of the inquiries had been placed at his disposal.

#### The Continental Price Agreement

For some years past there had been considerable competition on the Continent, which led to varying prices for similar products in different districts and countries. The position was also complicated owing to the sale of Reparation dyestuffs, where different methods of sale were employed in each of the allied countries. The competition of the three leading groups of makers on the Continent brought about low prices, particularly in France, Belgium and Holland. As was intimated by Dr. Brodbeck at the last meeting of the Society of Chemical Industry in Basle, an agreement had been entered into between the French, German and Swiss groups of colour manufacturers for the purpose of sales co-operation, with the result that the period of keen competition on the Continent, to which reference has been made, had now given way to a more stable and fairly well-defined price policy. This had had the effect of increasing prices in some countries, particularly in those where very low prices had hitherto prevailed.

This price convention would regulate the price of dyestuffs in Continental countries, and they believed that the cartels would have the effect not only of maintaining prices, but also of tending to increases in some instances. So far as could be ascertained, the agreement embraced only the leading Continental makers, but it was generally believed that arrangements would be made with certain makers in America which might be the prelude ultimately to a world-wide cartel. If such an eventuality materialised, it was to be hoped that the interests of the users in this country would be adequately safeguarded, and that British users would be in a position to buy dyestuffs not only as advantageously as any of their competitors, but with that complete freedom of choice and without any territorial restrictions.

#### The Results of the Price Inquiry

It was quite clear from the inquiry already mentioned that at the present time, both on the Continent and in England, the index figure for dyestuffs as against pre-war did not compare favourably with the general commodities price level. There might be many reasons for this anomalous position, and probably one was that the price of benzol, owing to the extended use of that commodity, was now very much higher than pre-war, affecting consequently the price of toluol, naphthalene and anthracene. The price of coal suitable for distillation was low compared with other commodities, but it seemed evident that benzol selling prices had no strict relationship to the primary costs, and that the prices were now regulated more or less by demand and by the price of petrol.

Another reason which most probably influenced the high price of dyestuffs was that the capacity for production throughout the world was considerably in excess of the consumption, and whilst rationalisation was taking place as rapidly as possible, it seemed evident that the reduced output was being called upon to bear a higher share of standing charges. Many countries had now established their own dyestuffs factories, each of which might or might not be working economically.

The prices now ruling for dyestuffs in this country were not substantially higher than those paid by users on the Continent;

in fact, in several Continental countries the prices were much higher. In only two countries was it found that the average prices were lower than in Great Britain, namely, Germany and Holland. There were, however, still certain types of dyestuffs, particularly vat colours, where British users were paying more than their Continental competitors. The disparity was growing less and less year by year, and he was hopeful that before long British prices for this important range of dyestuffs would compare favourably with those ruling in any other country.

A survey of the major chemical prices ruling on the Continent showed that, speaking generally, British prices compared not unfavourably. In his opinion, this was probably due to the greater prevalence on the Continent of fixed-selling-price conventions.

#### The Dyestuff Export Trade

The world consumption of colour was undoubtedly increasing, but the world capacity for production was still far in excess of the output. According to the monograph on the chemical industry submitted by the Association of British Chemical Manufacturers to the Economic Conference of the League of Nations, it was estimated that the world consumption of dyestuffs aggregated 154,000 tons in 1924. To-day it was estimated that the consumption was nearer 200,000 tons per annum, and the capacity for manufacture was probably double that figure. It was computed that Germany claimed 40 to 45 per cent. of the world consumption, the major part of which was for use outside Germany. The German exports of dyestuffs and intermediates in 1927 were 72,616 tons as against the British exports of 3,882 tons. In 1927 France exported 7,212 tons and Switzerland 9,988 tons. Even to the Colonies and Dependencies, the British exports are not increasing as they should. In 1927, according to the *Zeitschrift für Farbenindustrie*, the exports from Germany increased considerably over 1925 and 1926, as the following table showed:—

	1925	1926	1927
	In Millions of Marks.		
Total exports from Germany .....	277.6	293.2	337.0
of which the following are the more important:—			
British-India .....	19.6	22.0	39.0
China .....	32.7	35.4	33.9
Great Britain .....	13.4	20.4	24.7
Holland .....	17.9	18.0	20.4
Czecho-Slovakia .....	16.8	16.4	20.1
U.S.A. .....	16.4	16.4	16.0
Japan .....	12.3	16.5	11.5
Russia .....	32.8	28.2	10.4

#### Room for Expansion

These figures demonstrated conclusively that there was room for a vast expansion of the export trade of British colours. It was apparent, of course, that if this country was only in a position to offer similar types of colours to those made in Germany, Switzerland or America, they would be in a difficult position to extend their exports, unless they could offer better value and service. At the same time, when they were without the fostering aid of protection, how could the British output be produced as economically as in those countries where the manufacture, in consequence of large export business, was on a much bigger scale?

The only true solution to sound economic production in this country would be found when it could take its place in the world's markets, and notwithstanding the moderate increases shown year by year, there was no definite evidence yet that British dyestuffs were being sold in the world's markets on a strictly competitive basis. From the information which had been made available to him in regard to convention prices ruling abroad, he ventured to suggest that there was considerable scope for British makers to obtain a footing in many foreign markets, particularly in the newer types of colours for acetate silk. Knowing as he did the capable export sections of I.C.I., he hoped it might not be thought presumptuous on his part to suggest that the time had now arrived when their dyestuffs section should be in the forefront of the export business, just as they were in the other sections of their industry. There was one market in particular in which it would be gratifying to see substantial increases; he referred to India.

The suggestion that Great Britain should form an organisation to determine colour tendencies on the lines of the Textile

Color Card Association of America seemed to him to be worthy of the support of the Association.

We were now faced with a critical period in the history of our dyestuffs industry. The Dyestuffs (Import Regulation) Act, 1920, would expire in January 1931, and steps were now being taken through the Dyestuffs Industry Development Committee to ascertain the considered views of both makers and users. It might be useful to reiterate the essential principles from the point of view of the colour users. It had been affirmed on many occasions that:—(a) The colour users of Great Britain must be assured of free access to the best quality and range of dyestuffs produced anywhere; (b) They must be on as favourable terms as regards prices as any of their world competitors; (c) The provisions of the Dyestuffs (Import Regulation) Act, 1920, as set forth in Section 5, Sub-section 1, shall continue in force for a period of ten years from the commencement thereof, and no longer.

Throughout the past eight years, consumers of colour had contributed very largely towards the establishment of the dyestuffs industry, but happily that period was now passing, and he believed the industry was rapidly approaching a sound economic basis when it could face world competition. They had Lord Melchett's own assurance to that effect in the public statement which he made some time ago:—

"I can state emphatically that I do not consider the British chemical industry to be in any way inferior to the German industry. . . . Further, our costs of production of chemical products are as low as, if not lower than, in other countries."

Their evidence of the prices ruling on the Continent was

corroborative of British efficiency, and he was glad to be able to say so publicly. He thought these views might be taken as a faithful representation of the official policy of the Colour Users' Association. Nevertheless, his personal opinion was that it would be economic folly to allow this great industry—so necessary for our trade security and now well on the way to being thoroughly established—to decline. The cost of bringing the industry to its present stage had been no mean one, and at the final lap it would be a serious blow to our prestige and to our overseas trade if anything were to arise now which might counteract the efforts of the past eight years. He regretted that no response had been made to his gesture of a year ago, that the makers and users should meet in a round table conference to discuss the best means of dealing with the situation which would arise at the expiry of the Dyestuffs (Import Regulation) Act, 1920, in January 1931.

The situation called for an intensive effort on the part of all interested in the general well-being of our industry, so that they should not be faced with a repetition of the parlous position of 1914. He had confidence that they were now entering on that new era so often foretold in the chemical world. What had recently been done in the production and sale of nitrogenous products might be taken as an indication of the forward policy of those two great chemical combines, Imperial Chemical Industries, Ltd., of Great Britain, and the Interessen-Gemeinschaft of Germany. There they had a rationalised industry, with prices regulated on the basis of efficient production and at such a level as would ensure a ready consumption. Was it too much to expect a similar ideal in the dyestuffs world?

## Dyes and Their Application: Recent Technical Progress

By L. J. Hooley

THE enormous scale on which gas reactions are now carried out in the inorganic field in connection with fertilisers is not yet paralleled on the organic side, although development is taking place rapidly. In this connection, the fundamental organic intermediates such as alcohols and benzene are of most interest, and the recent synthetic production of methyl alcohol is an outstanding indication of what is likely in the future.

Among those organic intermediates which may be regarded as of interest mostly for dyestuff purposes, the compounds are in many cases rather too complex, nor are they used in sufficiently large quantities, for gas methods to have been of much interest so far. The most important exception to this generalisation is, of course, phthalic anhydride, which is very important for the manufacture of eosins, galleins, rhodamines, indigo and anthraquinone derivatives.

### Manufacture of Phthalic Anhydride

Phthalic acid was first obtained by Laurent by oxidising naphthalene with nitric acid. This method, with variations in the oxidising agent, has always been employed since. When the requirements for indigo became very large, Sapper's process with mercury and sulphuric acid came into use (about 1896), and enormous quantities are understood to have been made in this way by the B.A.S.F., while incidentally the necessity for utilising the large quantities of by-product sulphur dioxide had an important influence in the development of the contact process for sulphuric acid.

The latest method, air oxidation, is associated with the names of Conover and Gibbs and their collaborators (in the Colour Laboratory of the U.S. Department of Agriculture); Wohl; and Weiss and Downs (of the Barrett Co.); and others in its later stages.

### Vapour Phase Oxidation

This work on vapour oxidation may be regarded as arising out of earlier work of Orloff on the vapour phase oxidation of hydrocarbons, and of Woog and Gibbs on the oxidation of toluene to benzaldehyde. Gibbs had already obtained successful results with toluene, using vanadium pentoxide and molybdenum trioxide as catalysts, and was thus encouraged to try the oxidation of naphthalene when none of the other methods of making phthalic anhydride examined promised to be altogether satisfactory for the newly-arising American dyestuff industry.

A great deal of preliminary work was necessary in order

to find suitable apparatus and the best catalyst, and to establish conditions such as temperatures and the proportions and rate of flow of air and naphthalene vapour. Phthalic anhydride is only one of a series of oxidation products of naphthalene, terminating in carbon dioxide and water, and including quinones, aldehydes, etc., so that selected conditions are necessary to ensure that phthalic anhydride is not only the main product but the only one produced in substantial proportions.

### Outline of the Method

Very briefly, the process consists in vapourising the naphthalene and passing this with hot air through the converters, where the mixture comes into contact with the vanadium pentoxide or other catalyst and oxidation takes place. The gaseous products pass on to the condensers, where the phthalic anhydride and unchanged naphthalene crystallise out, along with smaller quantities of by-products (see Fig. 1). The various crystal fractions are separated as far as possible, and further purified by re-sublimation or analogous methods. The phthalic anhydride could, of course, be easily and completely separated from the naphthalene by alkali extraction; the resulting product would then, however, be sodium phthalate, which for most purposes would need to be converted first to the free acid and then to the anhydride, but this would add materially to the cost.

In actual manufacture, much of the difficulty which arises is in connection with plant and control, and for small-scale production this is likely to be too great to make the process economic. The method is being worked in America, by the Kuhlmann concern in France, and has now been run successfully for several years in this country by Scottish Dyes, Ltd.

During recent years considerable further work has been done on this and other analogous vapour phase oxidations. While some of this work deals with extensions on the chemical side, including the application of the process to new compounds or reactions, a good deal of it is concerned with catalysts and with plant design; much of it is of a tentative nature and will not find ultimate application.

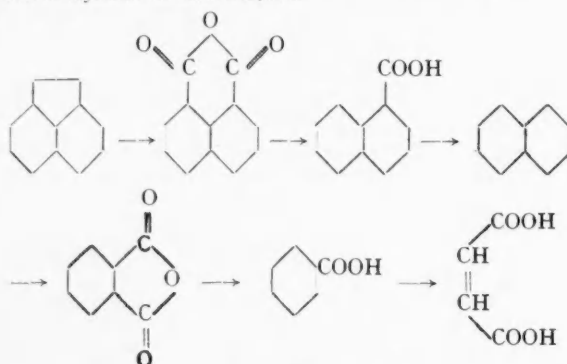
### Vapour Phase Synthesis of Dyestuff Derivatives

In U.S.P. 1,591,619 (H. D. Gibbs and the Du Pont Co.), alkyl naphthalenes are stated to be oxidised to phthalic anhydride in the same way as naphthalene itself. As a result of this, it is claimed that cruder qualities of naphthalene

may be used in the manufacture of phthalic anhydride than is usually the case. In U.S.P. 1,363,856 (Barrett Co., A. E. Craver), a catalyst consisting of 89 per cent. of uranium oxide mixed with 11 per cent. of molybdenum oxide is stated to be the most effective one for the oxidation of anthracene to anthraquinone. This patent contains considerable information on the behaviour of various metallic oxides as catalysts in this particular oxidation.

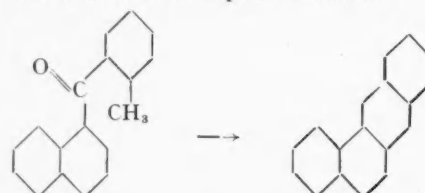
U.S.P. 1,648,833 describes oxidation of acenaphthene to acenaphthylene, using manganese dioxide as a catalyst. Naphthalic anhydride may be produced by the same process, yields as high as 90 per cent. being claimed. Substituted hydrocarbons such as nitro derivatives may be oxidised in

benzoquinone, maleic anhydride, acrylic acid, ethylene, etc., are instanced (see below). Another patent dealing with this decarboxylation is B.P. 262,101.

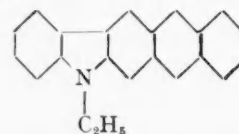


#### Ring Closure

Ring closure in the gas phase is described in B.P. 251,270, where aromatic ketones with methyl or methylene substituents in ortho-positions are passed over highly porous bodies at raised temperatures in the presence or absence of additional catalytically acting substances. As an example *o*-toluyl-naphthalene is converted to naphthanthracene.



A similar reaction in the heterocyclic series is given in B.P. 258,553 (I.G.F.A.-G.). Thus 3-*o*-toluyl-*N*-methyl-carbazole is converted to products amongst which is *N*-ethyl-naphtho-carbazole.



Of interest is the production of benzene from carbazole and naphthalene, and the conversion generally of cyclic compounds into analogues containing fewer rings. This is effected by treatment between 300° C. and dull red heat with hydrogen in the presence of a dehydrogenating catalyst. In this way benzene and aniline may be obtained from carbazole, or benzene from naphthalene (B.P. 283,600, I.G.F.A.-G.).

Maleic anhydride has, of course, been obtained in large quantities from benzene. It may also be obtained from furfural, as in B.P. 297,667 (Boehringer), which recommends for this purpose a considerable excess of oxygen or air.

#### Catalysts

It will be already noticed from the examples quoted above that a considerable variety of catalysts may be employed, according to the process which is to be carried out. For oxidation, the vanadium compounds, particularly the pentoxide, are the ones which have so far found greatest application. No doubt in some cases the alternatives have only been discovered in attempts to avoid already patented processes. Apart from the actual chemical nature of the catalyst, its physical condition and the medium on which it is deposited are also of importance. The actual production of vanadium pentoxide for use as a catalyst, and its purification, have been the subject of various patents. In U.S.P. 1,463,206 (Du Pont and H. D. Gibbs), infusorial earth is impregnated with molten vanadium oxide, cooled and broken up. This product does not fuse as easily as the oxide alone, it is not easily friable, and is convenient for coating bricks and other surfaces; the activity is also sometimes increased. In B.P. 153,877 (Barrett Co., C. R. Downs), the catalyst is deposited on



FIG. 1.—CRYSTALS OF PHTHALIC ANHYDRIDE IN A CONDENSER AFTER COOLING THE PRODUCTS OF THE VAPOUR PHASE OXIDATION OF NAPHTHALENE.

some cases without loss of the substituents. Thus *o*-chlorobenzoic acid may be obtained from *o*-chlorotoluene, nitronaphthalene from nitrophthalic acid, and nitroanthraquinone from nitroanthracene (B.P. 237,608, Maxted E. B. and Coke, B. E.).

In addition to producing carboxylic acid groups by the oxidation of rings and side chains, methods of removing carboxylic acid groups have also been detailed. Thus, by removing one carboxylic acid group from phthalic acid benzoic acid is obtained. In U.S.P. 1,645,180 (Monsanto and C. Conover), this is done by passing phthalic anhydride with superheated steam at a high temperature over a catalyst such as zinc oxide. Somewhat similar is B.P. 291,326 (I.G.F.A.-G.), while in B.P. 268,778 (I.G.F.A.-G.) oxidation and decarboxylation are carried out simultaneously, and the process may be combined with other catalytic reactions such as hydrogenation, dehydrogenation and hydration. The conversion of acenaphthene to naphthalic anhydride, naphthoic acid, naphthalene, phthalic anhydride, benzoic acid, benzene,

aluminium—for example, by vapourising on to small particles of aluminium or by melting with aluminium and stirring while cooling, so as to obtain a large number of comparatively small particles with rough and jagged surfaces.

Catalysts may be mixed with alkaline metals or alkaline earth salts and other substances to give products whereby more accurate control of the reaction may be obtained, especially in processes where more than one stage is involved. These salts are described as having a stabilising effect on the catalyst (B.P. 291,419, Selden). In B.P. 298,142 (Monsanto), vanadium is used in the form of solid complex silicates, these being made in the wet way by reaction between a suitable vanadium compound and a dissolved or gelatinous silicate. Zeolites described as "multi-component" zeolites are used in B.P. 296,071. Another point of interest is the use of the catalyst in the form of vapour or fine smoke (B.P. 262,201, Gaspari).

#### Control of the Catalyst

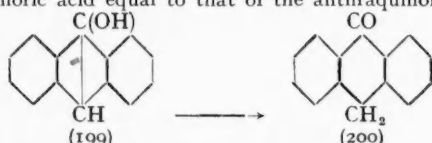
Perhaps the most important feature of all these processes is accurate control of the conversion process, which resolves itself largely into accurate control of the conditions in the neighbourhood of the catalyst. Assuming that all troubles in connection with the poisoning or deterioration of the catalyst have been overcome or reduced to a practical basis, there is still the important factor of temperature control. This is most pressing in the oxidation processes. Here the reaction is attended by very large heats of reaction; this heat very easily raises the temperature, so that a further stage of oxidation is reached, resulting, of course, in still further heat development, so that oxidation may go as far as carbon dioxide or even result in ignition. It will, therefore, usually be necessary to provide some method for removal of excess heat from the neighbourhood of the catalyst. Many ingenious devices have been recently produced to deal with this matter

## Basic Intermediates for Dyestuffs: No. XXVIII.—The Anthranol Series

By "Consultant"

ALTHOUGH anthranol does not find any use in the dyestuffs industry as such, it leads to a very interesting and useful series of compounds, chief among which is the condensed ring system known as benzanthrone. Benzanthrone, through the intermediate stage of its monochlor derivative, will give *iso*-violanthrone, the dichloro derivative of which is Caledon Brilliant Purple RR, whilst the bromine substitution product gives us a deep violet vat dye. An amino*iso*violanthrone is used under the name of Viridanthrene B, and is capable of giving, on caustic fusion, Idanthrene Dark Blue BO, from which it may be seen that the series is of no inconsiderable value to the dyestuffs industry.

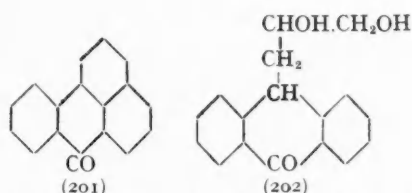
Anthranol itself is prepared by the reduction of anthraquinone by means of various reducing agents. In the older processes, it was customary to use zinc dust and caustic alkali, although the stereotyped laboratory reducing agent—tin and hydrochloric acid—has been quite successfully applied. In the latter process, the anthraquinone was dissolved in the least possible quantity of glacial acetic acid, and treated with two and half times its weight of tin, followed by a quantity of hydrochloric acid equal to that of the anthraquinone. The



mixture was raised to the boiling point, and further quantities of hydrochloric acid added until a filtered sample remained clear on cooling. On pouring the reaction mixture into dilute hydrochloric acid with stirring, the anthranol separates and may be filtered off. By this process, the yield amounts to 80 per cent. of the theoretical quantity, but at the present time anthranol is made almost exclusively by the reduction of anthraquinone with aluminium. The anthraquinone (20 lb.) is dissolved in concentrated sulphuric acid (3 cwt.), and the solution being stirred vigorously, aluminium powder (5 lb.) is added at such a rate that the temperature does not rise above 30-40° C. The reaction is completed when the temperature commences to fall, at which point the mixture may be run into water and the anthranol removed by filtration. Anthranol (199) forms a white microcrystalline powder which melts at 165-170° C., and is thereby partly changed to the isomeric substance anthrone (200).

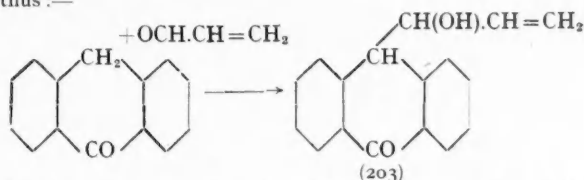
#### Benzanthrone

There are two distinct processes for the preparation of benzanthrone (201) itself. In the first of these, the starting material is crude anthranol. This substance (20 lb.) is dissolved in sulphuric acid (3 cwt.; 62 Bé.) and the solution stirred with glycerol (20 lb.). The temperature of the mixture is raised to 120° C., when oxidation takes place with the evolution of sulphur dioxide and the formation of benzanthrone. The process takes four hours for completion, after which the acid solution is run on to ice, and the greenish flocks of benz-



anthrone filtered off. Excess of anthranol and other impurities are removed by boiling the moist material with twelve times its weight of 1 per cent. soda solution.

The mechanism of the formation of benzanthrone in this manner is somewhat obscure, and has been the subject of considerable investigation. The first step is undoubtedly the conversion of the anthranol into anthrone, followed by condensation of the methylene group of anthrone with the terminal group of the glycerol, to form the intermediate compound (202). Alternatively, it has been suggested that the active agent is not glycerol itself, but allyl aldehyde obtained by decomposition of the glycerol with sulphuric acid. Under these circumstances the reaction would proceed thus:—



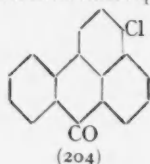
the intermediate compound (203) suffering ring closure, loss of water, and oxidation to benzanthrone.

In the second process for the production of benzanthrone, the preparation of anthranol is avoided. The starting point is anthraquinone, which is dissolved in forty times its weight of sulphuric acid (82 per cent.), and aniline sulphate slowly added (twice the weight of the anthraquinone used). This mixture is warmed to 100° C., and glycerol run in quantity equal to the anthraquinone. The process is thereupon conducted as previously described, the benzanthrone being separated by pouring into water. Further, as is more usual, the process can be adapted to use anthracene as its starting point. The anthracene ( $\frac{1}{2}$  cwt.) is dissolved in sulphuric acid ( $\frac{1}{2}$  ton; 62 Bé.) and glycerol added (1 cwt.). The mixture is heated for several hours at 100-110° C. until the anthracene has completely disappeared, at which stage the reaction mixture is allowed to cool and poured into water, the benzanthrone separating in brown flocks.

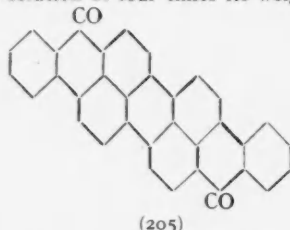
#### Violanthrone Derivatives

When benzanthrone is treated with chlorine, the latter enters the position indicated in the chloro derivative (204), and this intermediate affords the simplest method of synthesising the *iso*violanthrone ring. Benzanthrone is dried and ground

very finely, suspended in glacial acetic acid, and treated with chlorine at 100° C., when it becomes quantitatively converted



to the monochloro-derivative. This is converted into *isoviolanthrone* (205) by the following process: Chlorobenzanthrone is added to a solution of four times its weight of potassium



hydroxide in five times its weight of alcohol, and the temperature of the mass raised to 150° C. for several hours. The *isoviolanthrone* may be separated by treatment of the caustic mass with water, followed by the blowing of air through the

liquid to complete oxidation. The *isoviolanthrone* separates in deep coloured flocks, which are boiled with water, pressed and dried.

The dichloro-derivative of *isoviolanthrone* (Caledon Brilliant Purple RR) is obtained by chlorinating the base with sulphuryl chloride. The process is a comparatively simple one; the *isoviolanthrone* is dissolved in ten times its weight of nitrobenzene, and twice its weight of sulphuryl chloride added. The chlorination takes place quietly at 50-60° C., and the dichloro-compound, which is almost insoluble in nitrobenzene, is isolated by filtration, and washing with nitrobenzene, alcohol and water. The formation of the bromo-compound is a little more difficult to accomplish, and requires more drastic measures. The *isoviolanthrone* is heated with bromine in an autoclave at 150° C., when the monobromo-derivative is formed almost exclusively.

#### Viridanthrene B

The nitration of *isoviolanthrone* proceeds quite normally to the mononitro-compound, analogous to the monobromo-compound just described. The *isoviolanthrone* is dissolved in sixteen parts of glacial acetic acid, and nitric acid run in. Three parts of nitric acid (s.g. 1.47) are required. The mixture is heated to 60° C. and stirred therefor twelve hours. The reduction to the amino-body (Viridanthrene) is accomplished by stirring one part of the nitro compound with 60 parts of 10 per cent. sodium sulphide solution for three hours at 100° C. On pouring into water, the amino-compound separates and may be removed in the usual way.

## Dyestuffs Markets: The Month's Business in Review

### From Our Own Correspondents

#### Lancashire

In his address to the Colour Users' Association at its annual meeting on July 5, Sir Henry Sutcliffe Smith drew attention to the progress still being made in the British dyestuffs industry, and mentioned as events of outstanding importance the production of Caledon Browns G and R (I.C.I.), the equivalents of the fastest vat browns of the Indanthrene series. He also noted the introduction of the "6B" brands of Acid Violets by L. B. Holliday, and of Solway Blue R (I.C.I.), the equivalent of the widely used acid wool colour Cyananthrol R. Sir Henry stressed the advances that have been made in Great Britain, particularly in the manufacture of vat dyestuffs, and pointed out that British dyestuff makers have realised the importance of the development of ranges of fast colours demanded by the textile trades for coloured effects of extreme fastness to light and wear.

The price reduction policy, initiated by I.C.I. in January of this year, does not appear to have reached the limit. There is still a tendency to lower prices, not so much in the sense of all-round reductions, but in the case of individual dyestuffs. It cannot be said that dye prices are stabilised as yet.

The Clayton Aniline Co. have introduced two useful colours in Viscose Blues R and G, for which it is claimed that they give much more level dyeing effects on uneven dyeing viscose than do the standard brands of Sky Blues.

Considerable attention is at present being paid by printers to the problem of producing discharge effects on dyed acetate silk. There are obvious technical difficulties in the production of white discharges on dyestuffs suitable for this material, but there is no reason to fear that collaboration between dye manufacturer and printer will not eventually result in a comprehensive range capable of giving satisfactory white and coloured discharge effects.

#### Scotland

Business in Scotland during the past month has remained fairly steady, although towards the end the unsettling effect of the holiday period began to make itself felt. Sales of dyestuffs have shown a slight increase during the last two months, compared with the months immediately preceding. Calico printers were rather busier at the end of the month than in the middle, and could still do with more orders. The dyeing trade has been dull, but the finishing houses have been rather better placed. A new amalgamation in the dyeing and cleaning industry is announced, Stevenson Brothers (Dundee), Ltd.,

dyers and cleaners, who have a large number of branches in Dundee, Aberdeen, Edinburgh, and the East of Scotland generally, having been purchased by the Associated Dyers and Cleaners, Ltd., so that they will now be in close connection with Pullars of Perth.

The report of the Woollen and Worsted Committee, appointed by the Board of Trade under the Safeguarding of Industry procedure in December last, is now available, and makes interesting reading. Although the rate recommended was only 10-15 per cent., it would no doubt have proved useful to manufacturers.

An unsuccessful attempt has been made to have linen included in the Government inquiry into the cotton industry.

The new vogue for colour in linen goods promises to be of value to the Scottish industry, and hopes of better results are held out, as the Fife and other manufacturers are particularly skilful in the dyeing of linen and damask.

The tweed trade has improved again, especially in the fancy lines, and very useful export orders have been booked for America and Canada as well as the East and the Colonies, America and Canada especially being good customers. Many of the home orders are also probably destined for export, and, in fact, without these export orders conditions would not be so satisfactory.

#### German Exhibition of Chemical Plant

An exhibition of chemical apparatus and plant will be held at Frankfurt-on-Main, Germany, from June 10 to 22, 1930. Many foreign countries will be represented among the exhibitors. The exhibition will be held in four large halls, which are housed in three main buildings. The main avenues in the exhibition bear the names of famous chemists—Liebig, Bunsen, Wöhler, Emil Fischer, Nernst, Ostwald, Baeyer, Willstätter, Goldschmidt, Raschig, and others. The first hall will contain scientific apparatus and instruments for laboratory use, technical measuring instruments, and also the postal department, press-rooms, and writing rooms. In the second hall will be found porcelain and stoneware and products of the ceramic industry. Machinery and appliances used in the industry of oils and fats are to be assembled in Hall 3, a section of which will be devoted to the chemistry of daily life, whilst in the fourth hall large technical apparatus and machinery used in chemical industry, together with complete exhibitions of plant and processes and also raw and other materials, will be found.





# Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.

## The Dyestuffs Report

No official information is available respecting the report on the dyestuffs situation on which the Development Committee is now engaged, but it is understood that good progress is being made with the work, and that the report may be available within a couple of months or so. It may be remembered that at the recent meetings of the A.B.C.M. there were suggestions as to approaching the Government on matters relating to the chemical industry. As the Dyestuffs Act expires in about eighteen months time, it is obvious that the Government must come to some decision as to whether the Act should expire on the appointed day or be continued in some modified form. No authoritative deputation can very well approach the Government until the report of the Development Committee appears, because, until then, no official return will be available relating to the range, output, quality and prices of British dyestuffs. Not only will these figures be of great interest, but the recommendations of the Committee, on which makers and users are represented, together with Government nominees, are certain to carry weight with the Government and in the industry. In one case where the matter has been discussed, it is stated that the report will deal "with the general effect of the Act on the unfortunate consuming industries." The word "unfortunate" is a little significant as showing what the hopes of the writer are. But the prosperity of the dyers' organisations hardly suggests misfortune.

## New B.D.C. Colours

Imperial Chemical Industries announce the production of three new British Dyestuffs Corporation colours—Solway Blue RS, Lake Scarlet GS, and Naphthalene Fast Navy H. Solway Blue RS is a new addition to the B.D.C. range of acid alizarine colours, and produces very bright effects as shown on cross-bred slubbing, woollen yarn, and botany serge with cotton effects. On account of its excellent dyeing and fastness properties, Solway Blue RS is recommended as eminently suitable for the dyeing of piece goods and yarns and as of interest also to the dyer of loose wool and slubbing. This colour can be used in combination with chrome dyestuffs. It is dyed from a sulphuric acid bath consisting of 1 per cent. dyestuff, 2 per cent. sulphuric acid, and 10 per cent. Glauber's salt crystals. The material is entered and the dyebath raised to the boil, dyeing being continued for thirty minutes, when a further addition of 1.2 per cent. sulphuric acid may be made to obtain maximum exhaustion.

## A Fast Navy Colour

Naphthalene Fast Navy H, a deep satisfying tone shown on hosiery yarn in 4 per cent. and 6 per cent. strengths, is the latest addition to the B.D.C. range of acid colours. Although of interest to wool dyers in general, it is said to be particularly suitable for the dyeing of wool hosiery and knitting yarns, especially in view of the increasing demand for a higher standard of fastness to washing in this trade. The fact that it possesses very good fastness to sea water will commend it to dyers of yarn for bathing costumes. It is dyed in the usual manner for acid colours, but acetic acid is recommended in preference to sulphuric acid.

## Lake Scarlet GS

This is a further addition to the B.D.C. range of water-soluble lake-making dyestuffs, and is commended to the notice of manufacturers of printing ink colours and paper surfacing colours. The outstanding feature of the colour is its good fastness to light as compared with ordinary acid scarlets. It gives the best results when precipitated on a slightly acid base with barium chloride. The samples shown illustrate the use of the dyestuff as a paper surfacing colour. Where 10 parts of the colour are used to 90 parts of China Clay the effect is pale. A deeper and stronger shade is produced by a bath consisting of 40 parts aluminium sulphate (18 per cent.  $\text{Al}_2\text{O}_3$ ), 16 soda ash, 20 Lake Scarlet GS, and 70 barium chloride. All water soluble reagents are used as 10 per cent. solutions, the dyestuff is used as a 2 per cent. solution, and the precipitation temperature is maintained at 70° C. throughout.

## Azoic Colours on Wool and Silk

From John W. Leitch and Co., Milnsbridge Chemical Works, Huddersfield, we have received a pamphlet showing the application of the azoic colours to wool and silk. Of the eight samples of piece goods on the pattern card, six show these colours in different combinations applied to all-wool delaine and union goods, the seventh to cotton warp and woollen weft, and the eighth to cotton and wool mixture, both warp and weft. The colours used to produce a wide scale of tones are Fast Orange Base FW3 and Naphthol JWL2; Fast Orange Base FW3 and Naphthol JWL4; Fast Scarlet Base FW3 and Naphthol JWL3; Fast Red Base FW4 and Naphthol JWL2; Fast Red Base FW5 and Naphthol JWL5; Fast Claret Base FW2 and Naphthol JWL5; Brilliant Fast Scarlet Base FW2 and Naphthol JWL3; Fast Red Base FW4 and Naphthol JWL3.

The firm's new processes for the application of azoic colours to wool and silk eliminate entirely the use of caustic alkali. The processes are covered by British Patents Nos. 283347 and 283838. Customers who purchase the bases, and the Naphthols JWL required to be used in conjunction with them under these patented processes, will be granted free licences for the use of the quantities of materials purchased. These licences apply only to the actual purchaser for his own use of the materials, and are not transferable. Particulars are given in certain cases of the preparation of the bath and the method of application. These illustrate the general method of procedure, but the details naturally vary according to the depth of shade required. Further information on these points is available for those who need it.

## How Dyestuffs are Made

In the current number of the *I.C.I. Magazine*, there is a humorously illustrated article on "How Some Dyestuffs are Made," which may be recommended to the writer of technical reports as an example of how to tell a technical story in a simple, and indeed, an entertaining, style. Speaking of the rather forbidding names of the substances used, the writer remarks: "There is reason in these silly-looking long words of the chemist. 'Tetramethyldiaminobenzophenone' is really a kind of shorthand, however ridiculous it looks at first sight. To be sure, it is not

often that this sort of name is used in conversation, and this compound, like others shown in the diagram, is usually known, after the German chemist who studied it, as Michler's Ketone."

"The purpose for which dyestuffs are to be used," it is pointed out, "governs the character of the various dyestuffs that are made, that is to say, it is not enough simply to produce a red, or a yellow, or a blue substance, and so on, but that these compounds must be *dyes*, i.e., must colour the fabric so that they will not wash out or fade, or in general, if they are to be used in this or in any other way mentioned, their nature and properties must be adapted to that use. It is to fulfil these very varying requirements that so many chemically different dyestuffs are made; they do not represent as many different *colours*. And so it is that the number of chemicals used in the dyestuff factory becomes so great. Incidentally, it is because the demands are so many and so variable, changing with fashion and with development in other industries, that so much research must be carried out. New dyes can rarely be made to order; they depend on discovery as well as on invention, and the production of still more new chemicals for use in making dyestuffs is itself of great importance."

### Increasing American Dye Imports

Figures just issued by the United States Tariff Commission on the importation of coal-tar dyes during the month of June disclosed the fact that for the first six months of this year 3,267,478 lbs. of foreign dyes were imported, with an invoice value of \$2,627,558, an increase in both value and quantity over imports for the same period of 1928. The detailed figures on June imports are published below:

	1929		1928	
	lb.	Value.	lb.	Value.
January	552,821	\$432,505	415,156	\$327,148
February	390,664	323,514	478,407	391,351
March	766,786	612,253	378,191	316,183
April	576,193	445,200	633,815	505,152
May	523,817	428,780	382,233	295,969
June	457,197	385,306	411,662	309,607
Total	3,267,478	\$2,627,558	2,699,464	\$2,145,410

The percentage from different countries is shown in the following table:—

	June, 1929	June, 1928	Jan.— June, 1929	Jan.— June, 1928
Germany	66.0	63.4	71.05	64.21
Switzerland	28.0	25.9	24.15	23.98
France	1.0	1.2	1.12	1.71
England	2.0	3.0	1.42	2.26
Belgium	1.0	2.5	0.72	2.96
Canada	1.0	2.0	0.98	2.29
Italy	1.0	1.4	0.32	1.93
Netherlands	—	0.6	0.24	0.66

### Fast Dye Imports to America

The growing demand for fast dyestuffs finds the American producer for the moment unable to meet the demand, with the result that the imports of fast dyes of late have increased and have caused a renewed agitation for stricter tariff protection against foreign competition. "As a dye producer" the *American Dyestuffs Reporter* states "the United States has advanced to an enviable position in the world's trade. It has been supplying American mills with most of the colours they use—with 96 per cent. of their needs, calculated on the basis of poundage consumed. But, in the last few years, a great change has been wrought in the dyeing industry. With the demand for faster fabrics there has begun a steady swing away from the cheaper bulk colours which have comprised most of a mill's dyestuff needs. The public demand for faster merchandise has been passed on to the American dye industry in an appeal from the textile mills for faster colours. This has meant

extensive laboratory research and the development of many new types of colours. The range of fast dyes made in the United States is not complete, and in many cases the consumption of certain colours is not yet large enough to repay the industry for its work in developing them. In the face of this condition, the prices on many colours cannot be reduced until heavier production is called for. Meanwhile, the competition from the foreign dyes has steadily increased in these faster, more expensive types. The dye import tables show this trend plainly from month to month, from year to year."

### Demand for "Adequate Protection"

"The situation" it is stated "from this aspect, becomes very clear. The American dye manufacturers have been called upon by the mills for faster colours in a wide range of shades. They have, therefore, been concentrating their research efforts upon the development of these types and their progress has been exceptionally rapid, as all consumers of American vat colours will readily admit. But the competition from imported fast colours has paralleled our industry's advances. All the endeavours of the dye industry to develop new colours and to reduce prices on their present range of fast dyes have been hindered by an increasing foreign competition. The Government's monthly figures on dyestuff imports disclose that for the first six months of this year, more than three million lb. of foreign dyes have been imported, a total considerably larger than for the same period last year. The imports for 1928 were 27 per cent. higher than for the previous year, and at the present time they form about 20 per cent. of our consumption by value. The bulk of these imports has been distributed among the fastest types of colours, or in precisely those dyestuffs most in demand to-day. Only by adequate protection can America's development of the fast colours be continued." This, of course, is the position of the manufacturer. And, as usual, the view of the American importer is exactly the opposite.

### Dyestuff Licences

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during July, has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—

The total number of applications received during the month was 671, of which 581 were from merchants or importers. To these should be added 20 cases outstanding on June 29, making a total for the month of 691. These were dealt with as follows: Granted, 627 (of which 599 were dealt with within seven days of receipt); referred to British makers of similar products, 41 (of which 30 were dealt with within 7 days of receipt); outstanding on July 31, 23. Of the total of 691 applications received, 629 or 91 per cent. were dealt with within 7 days of receipt.

### Professor Mullin's Visit

In connection with the development of courses in rayon and textile chemistry, dyeing and printing, Professor C. E. Mullin, head of the Division of Textile Chemistry and Dyeing in the Clemson College Textile School, is now in Europe engaged on a further study of these subjects. Last year, Professor Mullin obtained valuable information regarding the manufacture, handling, dyeing and printing of rayon, and he has devoted much of his time during the past winter to the development and expansion of the rayon courses in the textile school. France has for centuries specialised in the manufacture and finishing of the finest fabrics from silk, which has many properties in common with rayon; hence the value of a knowledge of silk methods in connection with rayon. For this reason, the professor will spend considerable time in the silk districts of France and Italy, and particularly in the Lyons district.

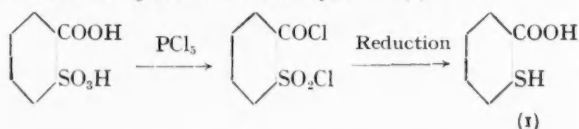
## Basic Intermediates for Dyestuffs: No. XXIX.—Some Thionaphthene Derivatives

By "Consultant"

THE thionaphthene derivatives find such an extensive use in the production of vat dyes of the thioindigo or semi-thioindigo class that no excuse is needed for the introduction of an account of their chemistry and production into this series. The introduction of a sulphur ring into aromatic compounds nearly always depends on the preliminary formation of a thiophenol derivative, in most cases a derivative of thiosalicylic acid, so that the production of this intermediate will be described first.

### Thiosalicylic Acid

It is only possible to prepare thiosalicylic acid on a large scale by the diazotisation of anthranilic acid, followed by the decomposition of the diazo solution with a concentrated solution of an alkaline hydrosulphide or of an alkaline xanthogenate. The reduction of the stable chloride of *o*-sulphobenzoic acid does give rise to thiosalicylic acid (1):—

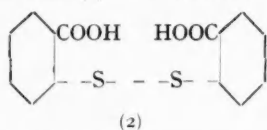


but the method, although satisfactory in itself, is of no value commercially.

A method which has been patented for the production of this acid is the treatment of orthochlorobenzoic acid with an alkaline hydrosulphide in the presence of copper powder or a copper salt. The method is said to work along the following lines: The orthochlorobenzoic acid (1 cwt.) is dissolved in the least possible quantity of strong caustic soda solution (about 86 lb. of NaOH are required), and the solution treated with potassium hydrosulphide (2 cwt.) and copper powder (1 lb.). The mixture is heated slowly to 150° C., and then raised to 200° C. fairly rapidly, at which point melting sets in. The temperature is then raised slowly again to 250° C., and the mass then gradually solidifies. On cooling, it is ground with water and dissolved in 400 gallons of the latter, and the thiosalicylic acid precipitated with dilute acid. The yield is good, but the inaccessibility of the raw material makes it very unlikely that this process will be used on anything like a large scale. Orthochlorobenzoic acid, it may be mentioned, may be produced from the orthochlorobenzaldehyde obtained as a by-product in the chlorination of nitrotoluene.

### The Anthranilic Acid Process

In the anthranilic acid process there is the disadvantage that dithiosalicylic acid (2) is the substance produced in the



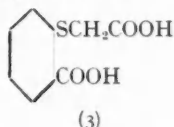
first instance when diazotised anthranilic acid is poured into a solution of an alkaline sulphide. This has to be reduced to the thioacid, thus introducing a complication which adds to the cost of production. The actual process is conducted by dissolving the anthranilic acid (1½ cwt.) in concentrated hydrochloric acid (2 cwt.), and diluting the solution with water (100 gallons.) The anthranilic acid must be ground to dust fineness to facilitate solution, which would otherwise not take place. Diazotisation is effected by running in a solution of sodium nitrite (70 lb.) in a little water, the temperature being kept below 5° C. The diazo solution is allowed to stand, and then run slowly into a sulphide solution prepared in the following way: Sodium sulphide crystals (2½ cwt.) are dissolved in water (40 gallons) and crushed ice (3 cwt.), and to the solution is added 40 per cent. caustic soda solution. During the addition of the diazo solution the temperature is not allowed to rise above 5° C., but after the addition it rises spontaneously to 15–20° C., at which point it is allowed

to remain until the evolution of nitrogen ceases. The dithiosalicylic acid is precipitated by the addition of hydrochloric acid, and removed by filtration.

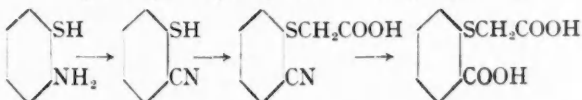
The reduction to thiosalicylic acid is readily brought about by iron powder in alkaline solution. The dithioacid (80 lb.) is dissolved in caustic soda solution (1 cwt. of 40 per cent. strength), and sodium carbonate added (½ cwt.) followed by iron powder (100 lb.). The whole is boiled until a sample gives on acidification no hydrogen sulphide, but a precipitate which is easily soluble in alcohol. The thiosalicylic acid may be precipitated by dilute acid. It may be added that it is desirable to filter the solution of dithiosalicylic acid from sulphur before it is used in this reduction. Thiosalicylic acid forms yellowish white needles, m.p. 164–165° C.

### Phenylthioglycolic Acid *o*-Carboxylate

The next intermediate of importance in the thioindigo series is phenylthioglycolic acid *o*-carboxylate (3). It is



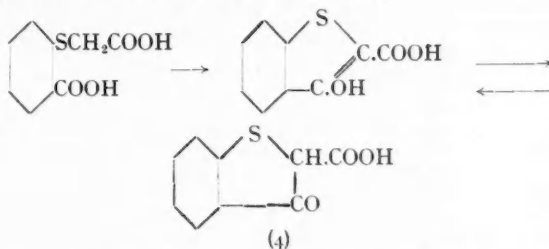
prepared on a very small scale by the action of thioglycolic acid on the diazo compound of anthranilic acid, but the yield is poor. It has also been prepared from *o*-aminothiophenol by conversion of the amino group to the cyano group, through the usual diazo and cuprous cyanide reaction, followed by treatment of the thiophenol group with chloracetic acid, the final stage being the hydrolysis of the CN group, thus:



This series of operations is commercially of little value, and the whole of the compound (3) used at the present time is prepared by the condensation of thiosalicylic acid with chloracetic acid. The thio acid (1½ cwt.) is dissolved in caustic soda solution (3 cwt. of 40 per cent. strength), and stirred with chloracetic acid (100 lb.). Condensation takes place readily, and the thioglycol derivative separates out after warming for an hour and making the resultant solution acid. It melts at 213° C.

### Thioindoxyl

When phenylthioglycolic acid *o*-carboxylate is subjected to a caustic fusion, ring formation takes place with the formation of the carboxylic acid of thioindoxyl (4).

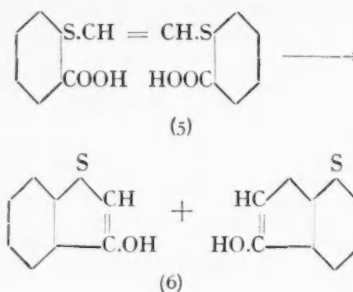


This reaction can be performed in open vessels, but a better yield is obtained when an autoclave is used. The phenylthioglycolic acid *o*-carboxylate is mixed with an equal weight of water and five times its weight of caustic soda, and the temperature of the mixture gradually brought to 170–180° C. It is maintained at this point for an hour, after which the cooled melt is dissolved in cold water and live steam blown in until no more carbon dioxide is evolved. This latter process converts the carboxylic acid of thioindoxyl into thioindoxyl itself; the latter substance is precipitated during

the reaction, and may be filtered off, or removed from the solution by means of superheated steam.

Alternatively, the cold melt may be dissolved in cold water and the thioindoxyl carboxylic acid precipitated by acidification with hydrochloric acid. The removal of carbon dioxide from this acid may be effected in various ways. The dry substance can be heated with acetic anhydride, or it may be suspended in paraffin or glycerol and heated to 200° C., at which temperature it is quantitatively converted into the thioindoxyl.

A very ingenious alternative method of preparing this substance is to heat thiosalicic acid with acetylene dichloride, a procedure which leads to the formation of the compound (5). This compound on heating with an equal weight of a mixture of equal proportions of caustic soda and caustic potash for half an hour at 220–230° C., gives thioindoxyl, which may



be removed in a pure condition by acidification of the melt, followed by distillation with superheated steam.

## German Chemical Society's Fastness Commission

### Examples of Fastness Tests (concluded)

In the fourth edition of the report of the German Fastness Commission, sections appear dealing with viscose and acetate rayon and setting out in detail the recommended test methods and standards of fastness. The tests given below (in continuation of those published in the "Dyestuffs Supplement" of June 8) deal with dyed acetate rayon.

#### Dyed Acetate Rayon

##### 1. Fastness to Light

THE test is carried out by exposing the samples, beside the types, dyed in yarn or piece, to daylight under glass in a cabinet located in the open, the materials being half-covered with thick paper.

Five types have been established:

I. One per cent. Methylene Blue BGX (659). The acetate rayon is mordanted in a 1:20 ratio bath, with an 8 per cent. solution of "Mordant for Acetate Rayon," for ten minutes at 50° to 60° C. After centrifuging, it is dyed, without rinsing, in a 1:30 bath for one hour, by entering at a low temperature and gradually increasing to 70° C., the bath containing 2 c.c. of acetic acid per litre.

II. Two-tenths per cent. Fuchsin A Powder (513). Mordanted and dyed as under I.

III. Three per cent. Cellit Fast Violet 4R. The material is dyed at a bath ratio of 1:30 for one hour at 60° to 70° C., with the addition of 50 per cent. calcined Glauber salt. After dyeing the goods are rinsed.

IV. Three per cent. Cellit Fast Violet ER, dyed with 50 per cent. ammonium chloride at a bath ratio of 1:30 for one hour at 66° to 70° C. Goods are rinsed after dyeing.

V. Ten per cent. Cellitone Fast Violet B Paste. Dyed for one hour at a bath ratio of 1:20, at 60° to 70° C., in a slightly frothing bath of 2 to 3 g. Marseilles soap per litre of distilled water. The goods are then rinsed in soft water.

##### 2. Fastness to Washing

The material is plaited with cotton, wool, silk, and acetate rayon, as given under 3, and treated for one-half hour at 40° C. in a bath ratio of 1:50, with 2 g. Marseilles soap per litre of distilled water, then squeezed out by hand, each handful being dipped each time into the bath, taken out, and squeezed. The material is finally rinsed and dried.

##### Standards.

I. Rather strong loss of depth and tone; white material coloured.

III. Depth and tone not altered; white material somewhat coloured.

V. No alteration in depth and tone; no bleeding upon the whites.

##### 3. Fastness to Water

The material is so plaited with cotton, wool, silk and acetate rayon that there are two parts of dyed material to one part of white material, of which some part of each kind must be in intimate contact with the dyed material. The sample is

immersed in distilled water for one hour, at about 20° C., at a bath ratio of 1:40. It is then squeezed and dried at the ordinary temperature. The treatment can be repeated for a second and a third time.

##### Standards.

I. With a single treatment the depth is altered but little; white material is strongly coloured.

III. With a second treatment (each time with fresh water), the depth is not altered; whites somewhat coloured.

V. With a third treatment (fresh water), the depth is not altered; the whites are not coloured.

##### Types.

I. Two per cent. Rhoduline Blue 5B, mordanted and dyed as given under I, I.

III. Ten per cent. Cellitone Fast Pink B Paste, dyed as given under I, V.

V. Four per cent. Cellitazol ST, developed with 4 per cent. Developer ON. The dyestuff is dissolved in hot water, any slight residue possibly present is dissolved by addition of a little acetic acid, and without any further addition the material is dyed for  $\frac{1}{2}$  hour at 40° to 70° C., at a bath ratio of 1:30; then rinsed, diazotized in the cold for 20 minutes with 4 per cent. sodium nitrite and 10 per cent. hydrochloric acid of 20° B., rinsed slightly, and developed for a half-hour at 60° to 70° C. with 4 per cent. of Developer ON, which has previously been dissolved with hot water and the same amount of caustic soda solution of 38° to 40° B. The developer before use is made slightly acid by addition of acetic acid.

##### 4. Fastness to Rubbing

The material is vigorously rubbed ten times, back and forth, with a piece of unfinished white cotton cloth stretched over the index finger. The extent of the rubbing should be about 10 cm.

##### Standards.

I. The dyeings rub off strongly.

III. The dyeings rub off somewhat.

##### Types.

I. Eight-tenths per cent. Methyl Violet B Extra (515), mordanted and dyed as given under, I, I.

III. Three per cent. Cellit Fast Violet ER, dyed as given under I, IV.

V. The dyeings do not rub off.

V. One-quarter per cent. Cellitazol B, developed with 1.5 per cent. of Developer ON. One part of Cellitazol B is brought into solution with 50 parts of boiling water and 2 parts of hydrochloric acid of 20° B. After about  $\frac{1}{2}$  hour's dyeing, 10 per cent. of sodium acetate is added, the dyeing completed in  $\frac{1}{2}$  hour, the material rinsed, diazotized, and developed as given under 3, V.

#### 5. Fastness to Ironing

The dyed material is covered with a thin, white unfinished cotton cloth, which is moistened with distilled water (100 per cent. humidity), and ironed with an iron which is at a temperature of 100° C. The temperature of the iron is determined by means of alpha-naphthol (m.p. 96° C.) and pyrocatechin (catechol) (m.p. 104° C.). The ironing is continued until the moist overlaid cloth is completely dry. The alteration of the dyeing is to be estimated by comparison of the still hot area with the adjacent unironed parts of the dyed material, and any marking off upon the overlaid white cloth is to be noted.

##### Standards.

I. Tone unaltered. Dyeing marks off badly upon the cotton cloth.

III. Tone unaltered. Dyeing marks off somewhat upon the cotton cloth.

V. Tone unaltered. Dyeing does not mark off.

##### Types.

I. Three per cent. Cellit Fast Blue A, dyed as given under 1, III.

III. Eight-tenths per cent. Methyl Violet B Extra (515), mordanted and dyed as given under 1, I.

V. Four per cent. Cellitazol ST, dyed, diazotized, and developed as given under 3, V.

#### 6. Fastness to Sulphur Steaming

The dyed material, plaited with wool and cotton, or sewed into a piece of union goods, is wetted out in a solution of 5 g. Marseilles soap per litre of distilled water, then wrung out. It is then hung up in a chamber filled with the fumes of burning sulphur, where it is left overnight, then well rinsed in cold water, squeezed, and dried.

##### Standards.

I. Depth and tone strongly altered.

III. Depth and tone little altered; white material somewhat coloured.

V. Depth and tone not altered; white material not coloured.

##### Types.

I. Five per cent. Metachrome Orange 3R Double, dyed as given under 12, I.

III. One per cent. Diamond Green BXX (495), mordanted and dyed as given under 1, I.

V. Four per cent. Cellitazol ST, dyed, diazotized, and developed as given under 3, V.

#### 7. Fastness to Perspiration

The test is to be carried out as follows:

The dyed material is laid out between layers of acetate rayon, unweighted natural silk, bleached cotton muslin, and woollen cloth, so that immediate contact is established with each, and the whole rolled up together, or the above materials are plaited together. The sample is placed in a solution of 5 g. of common salt and 6 c.c. of ammonia water of 24 per cent. per litre, bath ratio 1:10, left there for one-half hour at 45° C., and squeezed by hand ten times every ten minutes. After one-half hour, 7.5 c.c. of glacial acetic acid per litre are added, and the above treatment, with squeezing, continued for another one-half hour. The sample is then removed, squeezed, and dried at the ordinary temperature without rinsing.

##### Standards.

I. Depth and tone altered; strong bleeding upon the white material.

III. Depth and tone not or only little altered; white material somewhat stained.

##### Types.

I. One per cent. Methylene Blue BGX (659), mordanted and dyed as given under 1, I.

III. Ten per cent. Cellitone Fast Blue B, dyed as given under 1, V.

V. Depth, tone, whites unaltered.

#### 8. Fastness to Alkali (Street Dirt)

The dyed material is spotted with a solution of 10 g. of quicklime and 10 g. of ammonia water of 24 per cent. per litre, squeezed, dried at the ordinary temperature, and then well brushed.

##### Standards.

I. Tone strongly altered.

III. Tone somewhat altered.

V. No change in the colour tone.

##### Types.

I. One-half per cent. Rhodamine B Extra (573), mordanted and dyed as given under 1, I.

III. One-fourth per cent. Cellitazol B, dyed, diazotized, and developed as given under 4, V.

V. Four per cent. Cellitazol ST, dyed, diazotized, and developed as given under 3, V.

#### 9. Fastness to Cross Dyeing (Acid Boil)

The dyed material, sewed into white woollen cloth, is treated, at a bath ratio of 1:70, at 90° C., with a solution of 2.5 g. of sodium bisulphate per litre of distilled water, for 1½ hour, then rinsed and dried.

##### Standards.

I. Depth and tone rather strongly altered; white material strongly coloured.

III. Depth and tone hardly altered; white material slightly coloured.

##### Types.

I. Three per cent. Cellit Fast Blue A, dyed as given under 1, III.

III. One per cent. Cellitazol SR, developed with 1.5 per cent. of resorcinol. One part of Cellitazol SR is brought into solution with 50 parts of boiling water and 2 parts of hydrochloric acid of 20° B. After about  $\frac{1}{2}$  hour's dyeing, 10 per cent. of sodium acetate is added, the dyeing completed in  $\frac{1}{2}$  hour, the material rinsed, diazotized with 4 per cent. sodium nitrite and 10 per cent. hydrochloric acid of 20° B. for 20 minutes in the cold, quickly rinsed, and developed for  $\frac{1}{2}$  hour with 1.5 g. of resorcinol (previously dissolved in hot water) at the ordinary temperature.

V. Four per cent. Cellitazol ST, dyed, diazotized, and developed as given under 3, V.

#### 10. Fastness to Cross Dyeing (Neutral Bath)

The dyed material, sewed into white union cloth, is treated for one hour, at bath ratio of 1:50, with 20 per cent. of calcined Glauber salt at 85° to 90° C., then rinsed and dried.

##### Standards.

I. Depth and tone strongly altered; strong bleeding upon white material.

##### Types.

I. One-half per cent. Cellit Fast Yellow GGN. The dyestuffs is dissolved in hot water, and the material dyed for 1 hour, at 70° C., at a bath ratio of 1:30, with the addition of 5 per cent. acetic acid of 30 per cent., and 30 per cent. of calcined Glauber salt; then rinsed.

III. One per cent. Cellitazol SR, dyed, diazotized, and developed as given under 9, III.

III. Depth and tone hardly altered; white material somewhat coloured.

V. Depth and tone unaltered; white material uncoloured.

V. Four per cent. Cellitazol ST, dyed, diazotized, and developed as given under 3, V.

### 11. Fastness to Acids

The dyed material is spotted with a 1:10 solution of tartaric acid, and the alteration in tone estimated by comparison with a part spotted with water, the test to be made in both the moist and the dry condition.

#### Standards.

I. Strong alteration in tone.

III. Slight alteration of tone.

V. No alteration of tone.

#### Types.

I. One per cent. Cellit Fast Yellow R, dyed as given under 1, III.

III. Five per cent. Cellitone Fast Red Violet R Paste, dyed as given under 1, V.

V. Four per cent. Cellitazol ST, dyed, diazotized, and developed as given under 3, V.

### 12. Fastness to Scrooping

The dyed material is worked for five minutes in a bath of 3 g. of tartaric acid per litre of distilled water, at a bath ratio of 1:30, at the ordinary temperature, and dried without rinsing. The valuation of the colour tone is to be made immediately after the scrooping.

#### Standards.

I. Strong alteration of the colour-tone.

III. Slight alteration of the colour-tone.

V. No alteration of colour-tone.

#### Types.

I. Five per cent. Metachrome Orange 3R Double, dyed for 1 hour at 60° to 70° C., in a bath ratio of 1:30, with the addition of 30 per cent. calcined Glauber salt, then rinsed.

III. Three per cent. Cellit Fast Red BB, dyed as given under 1, III.

V. Four per cent. Cellitazol ST, dyed, diazotized, and developed as given under 3, V.

### 13. Fastness to Chlorine

The test is made by two methods:

(a) The sample is plaited with the same amount of cotton, wetted out in warm water, and immersed for one hour, at about 15° C., in a freshly-prepared bath of sodium hypochlorite (1 g. of active chlorine per litre), then rinsed, soured, rinsed, squeezed, and dried.

(b) As given under (a), except that bleaching powder solution (1 g. of active chlorine per litre) is used in place of sodium hypochlorite.

#### Standards.

I. Treated according to (a) and (b); depth and tone strongly altered.

III. Treated according to (a); depth and tone not altered. Treated according to (b); depth and tone altered.

V. Treated according to (a) and (b); depth and tone not altered.

#### Types.

I. Four and one-half per cent. Cellitone Blue Extra Paste, dyed as given under 1, V.

III. One per cent. Cellitazol ORB, developed with 1.5 per cent. of phenol, dyed as given under 10, I, diazotized and developed as given under 9, III.

V. One-half per cent. Cellitazol B, developed with 2.5 per cent. of Developer ON, dyed, diazotized, and developed as given under 4, V.

### 14. Fastness to Bleaching

The sample is plaited with wool, cotton, and chappe silk in such a manner that two parts of dyed material are present for each part of the undyed, and the whole bleached with hydrogen peroxide. The bleach bath is made up with 100

parts of distilled water and 20 parts of hydrogen peroxide of 10 to 12 per cent. by volume, and this bath is made very weakly alkaline with ammonia water. The bath must remain weakly alkaline during the bleaching (test with red litmus paper). The prepared material is laid into the bath at about 40° to 50° C., at a bath ratio of 1:40 to 1:50, and left in the slowly cooling bath for twelve hours. Care must be taken to keep the material always submerged in the bath, and to avoid strong agitation. The samples are then rinsed and dried.

#### Standards.

I. Depth and tone strongly altered; white material strongly coloured.

III. Depth and tone little altered; white material somewhat coloured.

V. Depth and tone not altered; white material not coloured.

#### Types.

I. One per cent. Auramine O (493), dyed as given under 1, I.

III. Five per cent. Cellitone Fast Red Violet R Paste, dyed as given under 1, V.

V. Four per cent. Cellitazol ST, dyed, diazotized, and developed as given under 3, V.

### 15. Fastness to Milling

The sample is plaited with cotton, wool, and silk, in the proportion of two parts of dyed to one part of undyed material, and so that each kind of fibre is in immediate contact with the dyed material. The sample is then treated at 30° C., at a bath ratio of 1:40, with a milling bath of 20 g. of Marseilles soap per litre of distilled water. It is first well worked with the hand, then left in for two hours, then thoroughly milled again by hand, washed and dried.

#### Standards.

I. Rather strong degradation of the dyeing; white material rather strongly coloured.

III. Depth and tone somewhat altered; white material somewhat coloured.

V. Depth and shade not altered; no bleeding upon white material.

#### Types.

I. One per cent. Methylene BGX (650), dyed as given under 1, I.

III. Four and one-half per cent. Cellitone Blue Extra Paste, dyed as given under 1, V.

V. Four per cent. Cellitazol ST, dyed, diazotized, and developed as given under 3, V.

### 16. Fastness to Potting

The dyed material is sewed into white woollen cloth, and treated for two hours in a 1:70 bath of distilled water at 90° C., then rinsed and dried.

#### Standards.

I. Depth and tone strongly altered; white wool strongly coloured.

III. Depth and tone hardly altered; white wool only slightly coloured.

#### Types.

I. One per cent. Cellit Fast Orange G, dyed as given under 1, III.

III. One per cent. Cellitazol RB, developed with 1.5 per cent. of phenol. One part of the dyestuff is dissolved in ten times the quantity of cold formic acid of 85 per cent. After addition of boiling water, the dyestuff, which will have separated out in a finely divided state, is got into complete solution by addition of 0.8 litres of hydrochloric acid per 100 g. of dyestuff. The material is dyed in this bath without any other addition, then diazotized and developed as given under 9, III.

V. Depth and tone unaltered; white goods not coloured.

V. One-fourth per cent. Cellitazol B, developed with 1.5 per cent. of Developer ON, dyed, diazotized, and developed as given under 4, V.

## Dyes and Their Application: Recent Technical Progress

By L. J. Hooley

RESEARCH on sulphur colours is not extensive at the present time. The more important features of the last two years or so are as follows:—

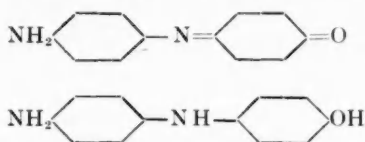
In the production of sulphur blacks from dinitrophenol, a process which gives an increased yield of 25 per cent. of Sulphur Black is said to consist of the treatment of the sodium salt of dinitrophenol with a polysulphide obtained by mixing 60 per cent. of sodium sulphide ( $\text{Na}_2\text{S}_{4.5}$ ), 28 per cent. of flowers of sulphur and 3 per cent. of crude sulphur.

New Sulphur Blacks from nitrophenols are described by Vidal (B.P. 283,468), the process consisting of submitting either the di- or tri-nitrophenol to the action of sodium sulphide and then cooling and adding nitrosophenol or nitroso *o*- or *m*-cresol, when a reaction begins in the cold and is completed by refluxing with an alkali polysulphide. The dyestuff is finally isolated by precipitating with an ammonium salt.

Vidal (T.I.B.A. 1926, 1,165) gives a general historical review of the Sulphur Blacks, outlining their methods of preparation and application and giving special attention to their properties as compared with aniline black. The usual methods employed in making blacks with dinitrophenol give products contaminated with sulphur, and this sulphur may cause tendering of the fibre. The author gives a method of his own discovery to avoid this formation of sulphur, and claims that the sulphur Blacks can thus be used to replace Aniline Black.

### Carbazole-Indophenol Products

One of the most important classes of intermediates for the synthesis of sulphur colours is the indophenol or leuco indophenol class, of general structure as shown below:



which are converted to dyestuffs by the usual sulphur melt. Where carbazole forms one of the aryl nuclei in the indophenol molecule, the dyestuffs formed after sulphur treatment belong to the important Hydron Blue class.

Among the improvements recorded in the dyes obtained from carbazole-indophenols and similar to the Hydron Blue type there may be mentioned that of B.P. 199,360 (S.C.I.B.), where the usual sulphide treatment of the leuco indophenol from carbazole and *p*-nitrosophenol is carried out in the presence of benzidine. Like Hydron Blue, the products dye indigo tints either by the sodium sulphide or alkaline hydrosulphite methods. A further improvement along these lines is given in B.P. 243,739 (S.C.I.B.), where derivatives of benzidine may be used instead of benzidine itself. Alternatively other organic bases may be employed, *e.g.*, aniline or naphthylamine. The treatment with sulphur may include the use of an alcohol as flux or of common salt as a diluent. As before, the products are blue, with redder shades in those cases where instead of the base itself an acetyl derivative is used in the melt, as, for example, diacetyl-benzidine. Greener shades are obtained in the same process if, in addition to the amine, urea is also added, as in B.P. 270,348 and U.S.P. 1,662,415 (S.C.I.B.). In a still further patent, B.P. 280,595, of the same manufacturers, the arylamine is omitted and the urea or other similar compound alone used; compounds mentioned are semicarbazide, guanidine, thiourea, acetamide.

Another improvement in the process refers to an earlier stage, namely, the production of the indophenol. Here the condensation of the carbazole or alkyl carbazole with the *p*-nitrosophenol is carried out at lower temperatures than previously, as, for example, in U.S.P. 1,637,868 (Du Pont), where *N*-ethyl-carbazole and *p*-nitrosophenol are combined below  $-130^{\circ}\text{C}$ ., and the product converted immediately to the stable leuco derivative by treatment with sodium sulphide. Somewhat similar is U.S.P. 1,662,062, where indophenols are produced by condensing the carbazole with an excess of *p*-nitrosophenol in sulphuric acid at, for example,  $-5^{\circ}\text{C}$ . (Nat. Aniline and Chem. Co.).

### Use of Hydro Derivatives

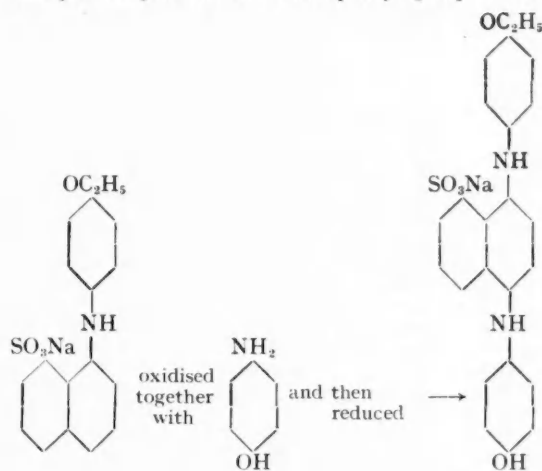
A still further innovation consists in the use of hydro derivatives as starting materials in the preparation of the indophenols. For example, in G.P. 443,685 and B.P. 286,005 (I.G.F.A.-G.), dihydro-indoles are used, these being converted to indophenols in the usual way, *e.g.*, by oxidising in the presence of *p*-aminophenol or condensing with *p*-nitrosophenol or with the quinone chlorimide. As a specific example, 1-methyl-2:3-dihydroindole is treated with *p*-aminophenol and hydrochloric and sulphuric acids, followed by oxidation with sodium bichromate; the leuco indophenol is then obtained with sodium hydrosulphite. Other examples include the products from *p*-aminophenol and 2:3-dihydroindole and 1-phenyl-2:3-dihydroindole. The indophenols or leuco indophenols may be condensed to sulphur dyes in the usual way.

An extension of the principle is seen in B.P. 285,382, where hexahydrocarbazole, *N*-ethylhexahydrocarbazole, tetrahydroquinoline, etc., are mentioned as suitable hydrogenated bases. The actual condensation to sulphur colours of these leuco indophenols is described in B.P. 297,123. As an example, the leuco indophenol from *p*-aminophenol and hexahydrocarbazole is treated with calcined sodium sulphide and sulphur in the presence of alcohol. The product dyes in bluish-violet shades. From octahydroacridine and *p*-aminophenol a green dye can be obtained.

The use of the ammonium salts in separation has already been referred to above in connection with Sulphur Blacks. They may also be used in connection with the isolation of indophenol products.

### Green Sulphur Colours

New green sulphur colours similar in general constitution to the Thionol Greens are described in B.P. 299,909 (I.G.F.A.-G.). Thionol Green, which may be regarded as having a complex Immedial Blue structure, is obtained by the sulphurisation of 1-arylamino-4'-hydroxyphenyl-4-aminonaphthalene-6 (or -7 or -8)-monosulphonic acid with sodium sulphide and sulphur. In the present patent, one of the phenyl groups contains

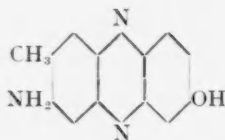


alkoxy groups in the *p*-position, as shown above. For example, one leuco-indophenol consists of 1-*p*-phenetylamino-4-*p*-hydroxyphenylaminonaphthalene-8-sulphonic acid. This leuco indophenol can be obtained in the normal way by oxidising a mixture of *p*-phenetyl-1-naphthylamine-8-sulphonic acid and *p*-aminophenol, and reducing the resulting indophenol. The dyestuff obtained according to this patent gives shades which are remarkable for their yellowish tinge.

### Phenazine Derivatives

Phenazine derivatives have already been used for making reddish-brown sulphur dyestuffs, for example, Immedial Bordeaux, where aminohydroxyphenazine is treated with sodium sulphide and sulphur. In a new patent, B.P. 299,152 (I.C.I., R. W. Pope and R. Wyler), instead of the usual

aminohydroxyphenazines, carboxylated derivatives of these are used.



For example, the product shown above is treated under pressure with sodium bicarbonate; on thionation it gives a reddish-brown dyestuff.

#### Sulphur Colours from Azo Chrome Dyes

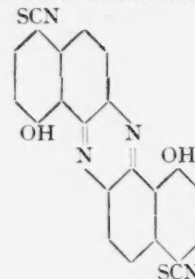
An unusual source of sulphur colours is given in B.P. 290179 (S.C.I.B.), where chromium compounds of azo dyes are used as starting materials, these being converted to sulphur dyes by the usual sulphurisation processes. For example, the chromium compound obtained by diazotising 1,2-aminonaphthol-4-sulphonic acid, coupling with alpha-naphthol, and afterwards treating with chromium formate, is refluxed with sodium sulphide and sulphur. A reddish-blue is obtained in this case. The dyes generally are stated to be fast to washing and light.

#### Thiocyano Derivatives

A new method of introducing sulphur into the dyestuff molecule is described in two patents, B.P. 240420 (A.G.F.G.) and B.P. 289241 (I.G.F.A.-G.). In the former, sulphocyno derivatives are obtained by adding lead sulphocyanide to sulphuryl chloride. On filtering off the lead chloride, a solution is obtained which is probably sulphuryl sulphocyanide. This solution will react with amino or oxyaryl compounds, for example with alpha-naphthol, which gives

4-sulphocyno-1-hydroxynaphthalene; and with diphenylamine it gives 4,4'-disulphocyanodiphenylamine.

These sulphocyno compounds are converted into thio-phenols or disulphides. As examples, dyestuffs are obtained from the sulphocyno derivatives of 8:8'-dihydroxy-1:2':1':2'-dinaphthazine and tolusafranine. The method is



applicable in cases where thionation does not take place by the usual methods.

#### Reserves Under Sulphur Dyes

A method of obtaining basic and azo reserves simultaneously under sulphur colours, described by Goubyrin in a sealed note, is examined in the *Bull. Soc. Ind. Mulhouse*, 1928, 367. The method depends on the fact that during steaming the formaldehyde compound of beta-naphthol will react with resorcinol under the influence of zinc chloride, giving on the one hand free beta-naphthol, which will couple with a diazo compound, and, on the other, a condensation product of resorcinol and formaldehyde capable of fixing basic dyes satisfactorily.

## Dyestuffs Markets: The Month's Business in Review

### From Our Own Correspondents

#### Lancashire

THE dyestuff market during July has on the whole been dull; the shadow of the dispute in the spinning and weaving sections of the cotton industry has been over the whole trade.

The outlook in the dyeing and finishing branches is not at the moment at all bright. There has been a feeling, based no doubt on the comparative success of the Bleachers' Association, the B.D.A., and other combines and firms, that the finishing end of the cotton industry was taking an excessive toll from the manufacturing. The *Manchester Guardian* now reports that the possibility of the Finishers assisting in the recovery of the trade by modification of prices and conditions is again being discussed.

If pressure is brought to bear on the allied trades of bleaching, dyeing, printing and finishing, to lower prices, it is likely that the reply will be that this could be made possible only by a reduction in wages in the trades mentioned. There are, indeed, rumours that the employees having put forward proposals for improved terms, including payment for holidays, the employers may reply with a counter-proposal for a cut in wages.

The outlook is not too bright, but employers and employed in the dyeing and finishing trades have a good record as to freedom from strikes and lock-outs, and there is no need to anticipate anything of this kind. In the meantime, the shortage of goods, both yarn and piece, is already affecting the dyer and finisher, and short time is being worked.

The holiday period has so far prevented the reduced consumption of colour from being felt by the dye manufacturer.

#### Scotland

At present conditions are, of course, somewhat disturbed as a result of the holidays. In the case of the majority of mills in the Glasgow district, these are now over, but in others they are still to come. Taking conditions generally, however, business has been fairly satisfactory, and the outlook, when normal working has been resumed, is considered to be fairly good, provided there are no set-backs. At the moment of

writing the outlook in the Lancashire cotton industry has darkened again, as the Operative Spinners' Amalgamation have rejected the recommendation that their Executive Council should be empowered to negotiate the best possible terms with the employers. The spinners, therefore, have now come into line with the weavers in completely opposing any negotiations on the basis of wages, so the prospect is worse than a fortnight ago. So far, the stoppage has not had a great effect in Scotland; there has, of course, been some diminution in business, and this must inevitably increase if the stoppage lasts.

In the woollen trade in the south of Scotland, the present time being between the spring and winter order seasons, looms have not all been kept running, but this is a condition which it is hoped will be remedied in the near future.

Sir Henry Sutcliffe Smith's review of the dyestuffs situation at the tenth annual general meeting of the Colour Users' Association on July 5 was even more interesting and informative than usual. From the point of view of the dyestuffs industry, perhaps one of the most satisfactory points was the statement that the prices of dyestuffs in Great Britain generally now compare favourably with those ruling on the Continent.

#### Production of Yellow Ochre in Spain

THE production of yellow ochre in Spain is confined solely to the Provinces of Almeria and Alicante. Some ochre deposits are found in the Province of Malaga, but the product is reported to be of inferior grade and has no commercial value. Government mining statistics for 1927 record the output of ochre in the Province of Almeria during that year at 420 metric tons. The same report stated that the only other section in which ochre was mined was the Province of Alicante, the production being 1,530 metric tons. Ochre mined in the Province of Almeria is exported in the natural state, and the total exports from the port of Almeria were 525 metric tons in 1927 and 251 metric tons during 1928. Ground ochre shipped to Malaga from the Province of Alicante and exported from Malaga amounted to 191,071 pounds.





## Dyestuffs Monthly Supplement

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### The Griess Centenary

ON Friday, September 6, there occurred the centenary of the birth of J. P. Griess, the discoverer of diazo-compounds and the founder of the azo-dyestuff industry. Apart from his great work as a chemist, Griess is particularly interesting in that he was for many years resident in this country, to which he came in 1858, at the instigation of Heinrich Caro, to the Royal College of Chemistry, in London, as assistant to Hofmann. (At that time, according to the statement of Professor H. Grossmann in the *Chemiker-Zeitung*, many German chemists came to England, then the chemical centre.) The discovery of the diazo-compounds is best given in the words of Griess himself. The following quotation is taken from Dr. J. C. Cain's book, *The Chemistry and Technology of the Diazo-Compounds*.

### The Discovery of the Diazo-Compounds

"Dr. Gerland," wrote Griess, "when working in the laboratory of Professor Kolbe at Marburg, investigated the action of nitrous acid on amidobenzoic acid at the request of Kolbe. Thus oxybenzoic acid was prepared, indicating a chemical change then considered of much importance. In like manner I investigated a means of converting picramic acid into oxydinitrophenylic acid,  $C_6H_2(NO_2)_2(OH)_2$ , but I obtained instead of the latter a compound possessed of such striking and peculiar properties that I at once concluded that it must belong to a completely new class of compounds. Analysis soon showed me that this peculiar compound had the composition  $C_6H_2(NO_2)_2N_2O$ . Naturally I soon submitted other amido-compounds in like manner to the action of nitrous acid, and obtained thus, in almost every case, the corresponding diazo-compound. But the circumstance to which I was indebted for my success in obtaining the diazo-compounds was that of the treatment of the amido-compounds with nitrous acid in the cold, whereas in the earlier experiments of Hunt and Gerland a higher temperature was always attained, and consequently no diazo-compounds could exist.

### The First Azo-Dyestuffs

"Having obtained these diazo-compounds, I then tried their action on all possible substances, among which of course are the numerous class of amido-compounds. I found that the diazo-compounds combine directly with these, forming frequently brilliantly coloured substances which dye animal fibres directly. The first colouring matter thus prepared by me, which I obtained in the years 1861-62, was benzeneazo- $\beta$ -naphthylamine. It was first prepared on the large scale, to the best of my recollection, in the years 1865-66 by Caro, who was then chemist in the works of Messrs. Roberts, Dale and Co., of Manchester. I first recommended the oxyazobenzene obtained by me for use as a colouring matter in 1866."

Griess continued his researches on diazo- and azo-compounds during his three years' residence in London as Hofmann's assistant, and also afterwards, while acting as chief chemist at the brewery of Allsopp and Sons, at Burton-on-Trent (in which capacity many of his later years were spent). Here, although occupied busily in the vast brewery, Griess found time in which to prepare a large

number of new diazo-compounds, and these were then handed over to his friend, Dr. R. Schmidt of Dresden, for analysis. Of this matter an amusing account has been given by Hempel: "The new compounds prepared by Griess were regularly sent in small packets from Burton-on-Trent to Dresden to be analysed. At the same time a welcome accompaniment took the form of barrels of Allsopp's finest Pale Ale!"

### New Colours

The Scottish Dyes, Ltd. branch of Imperial Chemical Industries has issued pattern cards dealing with several new colours. Alizarine Red S powder is a new addition to the I.C.I. alizarine range, and is an important colour for the wool dyer. Its chief use is on chrome mordanted wool, but it can also be dyed on aluminium and iron mordants. In all cases it gives shades of excellent fastness to light and milling. In conjunction with other mordant colours it is widely used for obtaining mixed shades, such as drabs and browns. In the patterns it is shown as 0.25 per cent. on alum mordanted wool; 1 per cent. on chrome mordanted silk; 0.5 per cent. on chrome mordanted slubbing; 0.5 per cent. on mordanted wool; 2.5 per cent. on alum mordanted wool; 2.5 per cent. on chrome mordanted wool yarn; 1 per cent. on chrome mordanted slubbing; and 1 per cent. on chrome mordanted wool.

### Duranol Reds and Scarlet

A second pattern card illustrates new Duranol products giving a variety of red, scarlet and claret shades. The three original Duranol Reds (2B, G and B Double) are also included. Of the new colours, bright bluish scarlet to red scarlets are produced by Duranol Scarlet 3B, Duranol Scarlet B, and Duranol Scarlet 2G; while duller mode shades are produced by Duranol Maroon and Duranol Claret B. All are dyed by the usual methods for Duranol colours, and can be mixed with each other and with other Duranols to produce combination shades. In fastness to light, they can all be classed as "very good" to "excellent," and in this respect they rank among the best Duranol colours. In other respects they have the same fastness properties as the other reds already on the market.

### The British Colour Council

The proposal to form a British Colour Council will, it is hoped, take practical shape at a meeting of representatives of the various interests concerned to be held on October 9. The subject has been under consideration for some time, and a committee has been at work on the preparation of a scheme to bring about the determination, co-ordination, and propagation of colour tendencies for the fashion and allied trades. A provisional scheme which will be submitted to the meeting has among its objects the obtaining and distributing of advance information about fashion colour tendencies and the eliminating of the speculative element from colour decisions. It is urged that the discovery of the correct colours for a given season is the surest way to make buying and selling easy for the dyer, the manufacturer, and the distributor, and the aims of the Council will be directed along these lines.

The organisers have no doubt that their proposals will be adopted at this representative gathering of British colour-using firms, and that a Council will be elected which will

include many names prominent in the textile industry. The Council will maintain offices and an organisation in London and will conduct researches as to the colours which fashion will choose in Paris and wherever such knowledge is to be found.

### **Dyestuff Licences for August**

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during August, has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—

The total number of applications received during the month was 620, of which 544 were from merchants or importers. To these should be added 23 cases outstanding on July 31, making a total for the month of 643. These were dealt with as follows: Granted, 596 (of which 552 were dealt with within seven days of receipt); referred to British makers of similar products, 21 (of which 19 were dealt with within seven days of receipt); outstanding on August 31, 26. Of the total of 643 applications received, 571 or 89 per cent. were dealt with within seven days of receipt.

### **Report on Boiler Explosion**

In accordance with the Boiler Explosions Acts, 1882 and 1890, there has been issued, from the Board of Trade Surveyors' Office, Liverpool, the report of a preliminary inquiry into an explosion from a vacuum drying stove which occurred on February 8 at the works of the Clayton Aniline Co., Manchester. Nobody was injured by the explosion. In the observations at the end of the report, it is stated that "in this case the boiler pressure was considerably in excess of the working pressure of the pan, and though a reducing valve was fitted at the steam inlet and a relief valve on the body of the pan itself, the steam inlet valve had to be used for regulating the steam supply to prevent overpressure. As the reducing valve failed to operate, at a time when the steam supply was suddenly increased in pressure by the coupling up of a higher pressure boiler, an explosion was inevitable.

"If the relief valve had been placed in the steam inlet pipe near the reducing valve, the arrangement would have been improved; these fittings should in such cases be of ample size as compared with the steam supply pipe and it is also necessary that they should be kept in good working order."

### **The International Dyestuffs Agreement**

Notes have recently appeared in the German Press with regard to the agreement arrived at some time ago between the I.G. and the French and Swiss producers of dyestuffs. It is stated that the United States Department of Commerce reports that the allocated percentages of exports are Germany 70, France 20, and Switzerland 10, while another version gives the percentages as 75, 17, and 8. It is believed that the home markets of the participants are protected. In the Far East, French products will be disposed of by the German organisation, while in Southern Europe France takes over the sale of German products (Switzerland also having some preference). Presumably, as regards Southern Europe, it is mainly a question of the Italian and Spanish markets.

### **I.G. and Scandinavian Dyestuffs Market**

Representatives of the I.G. and three dye producers from Basel recently held a meeting at Copenhagen, during which, according to reports, the position of the various participants in the Scandinavian market was discussed in detail. The local Scandinavian representatives of the various firms and organisations were also present at the meeting. Nothing definite can be learned of the object of the meeting or its results, but it is understood that

market division and competitive conditions were discussed in detail. The sale of the products of the I.G. in Scandinavian countries is centralised in a subsidiary company, A/S Anilin Kompagniet, located in Copenhagen, while the Swiss dye manufacturers are represented by local agents, Victor Rathjens and Georg Schönwandt.

### **Colour Radiation of Organic Compounds**

An investigation on the scattering of colour by organic compounds is being conducted by the Bureau of Standards at Washington in co-operation with the U.S. National Research Council. Dr. R. M. Langer, of the Council, is at work on this study in the laboratory of the Optics Division of the Bureau under the direction of the chief of the division, Dr. C. A. Skinner. When a substance composed of molecules having many atoms—the organic compounds, for example—is illuminated by light of a single colour, the substance scatters not only the incident colour but also other colours which are characteristic of the scattering substance as well as of the incident light.

### **Infra-Red Spectra**

This phenomenon seems to be a consequence of the present theories of physics. It promises to be a powerful tool for the investigation of the infra-red spectra of complicated molecules. The change in colour observed is intimately connected with the long-wave-length radiation in the infra-red which the substance is capable of absorbing or giving out. This infra-red radiation has long been known, but is still little understood. It is due to the vibrations of the atoms in the molecule with respect to one another and to their rotations about one another. Solid substances also have infra-red radiations, which are sometimes due to the vibrations of the molecules about their fixed positions in the crystal lattice of which the solid is composed. These vibrations give evidence of the forces between atoms and of the energy with which chemical compounds are bound. The scattering experiments will therefore be of importance to chemistry, and should be of value in the analysis of dyestuffs.

### **Swiss Industry Prospering**

The manufacture of dyestuffs continues to be the most prosperous of the industries in Basel, and the output, which has increased steadily in recent years, promises to show a still further increase during 1929. Statistics are not yet available for March, but for the first two months of 1929 Swiss exports were valued at 12,015,000 Swiss francs, compared with 11,708,000 Swiss francs for the months of January and February of 1928. Practically all of the dyestuffs exported from Switzerland are manufactured in Basel. The trade of Basel factories with Germany during the first three months of 1929 has not been so good as during the same period of 1928, but a considerable improvement is shown in trade with the United States, France, and Italy, and exports to Great Britain have shown no decrease. Exports of dyes from Basel to the United States during the March quarter of 1929 were valued at \$478,867, against \$355,775 for the first quarter of 1928.

### **Japanese Dyestuffs Expansion**

It is reported that the Mitsubishi Dyestuffs Co. is enlarging its factory, and will shortly commence manufacturing Malachite Green on a larger scale than before. It is stated that the manufacturing process has been perfected by experts of the Government Industrial Experimental Station at Fukuyama, and that the feature of their improvement is a very low price raw material, which can be utilised in the production of this dye. It is anticipated that the price of the dye will be 20 per cent. less than at present. The process of manufacture will not be patented. It is thought that this company will be able to produce sufficient Malachite Green to fulfil all Japanese demands.

## Fast Colours on Leather

### Some Suggested Lines of Future Development

The following is a paper read by Mr. M. C. Lamb, of the Leathersellers' Technical College, London, before a Conference of Curriers, Light Leather Tanners and Trade Chemists at the Leathersellers' Hall, recently.

PROBABLY at no period in the history of the leather trade has there been a greater requirement for fast colours than at the present. It cannot be denied that a very large amount of coloured leather on the market fails to come up to the standard of fastness that is demanded by the user. The standard of fastness attained by the leather industry has not kept pace with that which has been reached in the domain of textile products. Probably the guarantees of fastness to light and washing which are now obtainable in the majority of textile materials have unconsciously created a standard in the public mind that all coloured materials should be similarly fast. Whilst it cannot be disputed that some degree of improvement in the fastness of coloured leathers has been effected in the last two decades, the standard just referred to has by no means been attained.

#### Guarantees Suggested

Whereas it was possible prior to the introduction of mineral pigment finishes to dye leather reasonably fast both to light and to rubbing, there has been some falling away from the older standard since the introduction of pigment finishes. The suggestion recently made by the National Leather Goods and Saddlery Manufacturers' Association that leather dressers should guarantee their leather to be fast, and accept responsibility for any claims which might result through lack of this property, indicates very clearly the necessity that much greater attention should be paid to this question of fastness. Coloured leather is now employed for purposes of clothing, where even a greater standard of fastness of colour to all climatic influences becomes necessary.

The term "fast colour" is very comprehensive. One is inclined to associate in one's mind the term "fast" as applied to colour with freedom from liability of fading when exposed to light. This is not the only modern requirement expected of coloured leather. It is probable that fastness to wet rubbing is in many cases even more important than fastness to light. Fastness can be classified as follows: Fastness to light, rubbing, perspiration, wetting, alkali, acid and heat.

In the glove leather industry, and also to a lesser extent in respect of leather for clothing generally, a further standard has been attempted in the direction of fastness to washing. Other requirements may be required of leather manufacturers, but the two most important at the present day are fastness to wet rubbing and fastness to light.

#### Fastness to Light

Whilst fastness to light has always been a necessity in certain classes of leather, notably book-binding leathers, upholstery, fancy leather goods, etc., which are required to resist exposure to light reasonably, in order that the articles should retain their original colour, the more recent introduction of leather for clothing purposes, which may be worn under out-of-doors conditions of direct sunlight, demands a greater degree of fastness. It is, of course, obvious that in order to retain this important branch of the leather industry it is absolutely essential that no appreciable change of colour shall result when a leather garment is worn out of doors, even under direct sunlight conditions, for a reasonable period of time. This necessitates very careful and judicious selection of dyestuffs that have been previously tested with a view to ascertaining their comparative fastness under such conditions.

The writer has published from time to time lists of dyestuffs which have been tested for their comparative fastness to light, and it may, perhaps, not be out of place to include a list showing the relative fastness under daylight exposure of a few of the more important ones:—

#### Comparative Fastness to Light of Representative Dyestuffs on Sumach Tanned Leather

FUGITIVE.	MODERATELY FUGITIVE.
Acid Violet.	Auramine II.
Fast Violet.	Auramine O.
Eosine.	Resorcin Brown.
Erythrosine.	Milling Scarlet.
Methyl Violet.	Roccelline.
Acid Green.	Fast Red A.
Chrysoidine.	
Magenta.	

FAST.  
Orange II.  
Acid Orange.  
Soluble Blue.  
Acid Yellow.  
Acid Phosphine.  
Naphthalene Black.  
Saffranine.  
Rhodamine.  
Crocein Scarlet.

VERY FAST.  
Methylene Blue.  
Tartrazine.  
Quinoline Yellow.  
Citronine.  
Nigrosine.  
Induline.  
Alizarine Cyanine.  
Azo Yellow.  
Azo Geranine.  
Methylene Green.  
Acid Magenta.

Unfortunately, on account of the more general employment of catechol tannin materials in the tannage of many of the modern leathers, a further difficulty is met with by the discoloration which takes place on account of the leather becoming reddened and darkened by exposure to sunlight. In the case of East India tannages in particular, very considerable alteration in the colour may take place when subjected to moderate exposure. In order to obtain colours which will stand exposure it is a necessity that the tannage should be one that is comparatively unaffected by such conditions. By comparison with the standard which has been created in the public mind by the introduction of fadeless fabrics, the leather manufacturer labours under considerable disadvantages, inasmuch as no textile fabric discolours under atmospheric influence in the same manner as leather is liable to do.

#### Mineral Pigments and Lakes

The introduction of mineral pigment finishes upon leather should have been very helpful, inasmuch as the majority of mineral pigments are less liable to fading than coal tar dyestuffs. When the pigment finishes consisted almost exclusively, in the early days of their introduction, of a mineral earth of a ferric base, the colours were fast in this respect. When the pigment finish manufacturer was required to cover the whole range of colours of the spectrum, in the absence of mineral earths capable of producing bright colours such as scarlet, greens, blues and violets, he had of necessity to resort to the employment of coal tar colour lakes in order to fulfil this requirement. Unfortunately, the selection of these has, in many respects, been of a haphazard nature. Whilst it is true there are many coal tar colour lakes which are almost equivalent in fastness to light to the mineral earths, there are many which are, by comparison, very fugitive.

Many coal tar colour lakes are liable to change when exposed to daylight—as, indeed, are coal tar colours—the fading being brought about by reduction or oxidation of the constituent components of the dyestuff itself. Fading is brought about by light of varying wave-lengths, of which the more important, perhaps, are the ultra-violet rays, which in many cases possess the most powerful influence. There are some exceptions, however, to this rule, inasmuch as some coal tar dyestuffs, whether in the lake form or otherwise, change colour more slowly on exposure to ultra-violet rays than they do in natural sunlight.

The fading of colour of a pigment finish on leather, when it is compounded with a colouring matter which is sensitive to light, is liable to be more rapid than when the leather is dyed with a similar dyestuff, inasmuch as the coloured surface is presented to the light in a more flattened form, less penetrable to light itself, and, owing to the more opaque result thus obtained, the fading occurs on a thinner layer of the upper surface.

#### Results of Recent Work

Considerable work has been done with a view to investigating the fastness to light of various dyestuffs and dyestuff components when applied to different materials. There is, however, a requirement in respect of a standard method of testing. A comparatively recent publication dealing with an investigation of the fastness to light of lake colours is one that would appear to be applicable for the standardisation of fading upon leather; the classification of various dyestuffs and dye lakes under the five following groups:—

(i) No perceptible change after one month's exposure—June, July or August.

(2) A slight loss in depth or alteration in shade after one month's exposure.

(3) A distinct loss in depth or alteration in shade after one month's exposure.

(4) Similar to (3), but after only 14 days' exposure; and

(5) Colour practically bleached after one week's exposure.

In connection with the investigation of the question, representative lakes were agreed upon as standards under the classified groups:—

(1) Alizarin Red (alumina-calcium phosphate lake).

(2) Permanent Red 4B (calcium salt).

(3) Lithol Red R. (barium salt).

(4) Eosin YS (lead salt, barium sulphate base).

(5) Lakes of less fastness than No. 4.

The above classification should be of interest to the leather manufacturer, inasmuch as it shows the extreme variability, for example, of red lakes, and the necessity for a choice by the pigment-finish manufacturer of lakes which will come under the classification of No. 1, and show no perceptible change of colour after months of exposure to sunlight during the period of more intense light in this country—i.e., June, July, August.

Many cases have come before the writer's notice of complaints being made of fading where the leather had been dyed a colour which bore no possible resemblance to the ultimate finished colour, so that when fading of the pigment finish had taken place on the surface of the leather, the leather regained the original dyed colour and a very objectionable contrast thus resulted. If the leather had been dyed with a fast dyestuff of a colour which was comparable with the finished result, any fading of the pigment which might have taken place would not have been so readily noticeable.

#### Technical Requirements

As regards the more important technical requirements in the production of coloured leather fast to light, these may be summarised as follows:—(1) One of the first necessities in the case of a leather which is required to withstand reasonable exposure to sunlight is for the dyer to select a dyestuff which is the fastest possible obtainable; (2) the leather should be dyed as full a shade of colour as is practicable; (3) the pigment finish to be used for the finishing operation should be selected with as great a care as regards fastness to light as the dyestuff.

A difficulty which has arisen recently in the case of many finishes used, particularly upon clothing leathers, has resulted through an actual decomposition of one of the components of the pigment finish producing an effect which was, on account of its opaque character and lightened colour, associated with actual fading. In order to obtain the requisite elasticity of the finish demanded in the case of leathers for clothing, several manufacturers have been experimenting with the addition of rubber latex. This product imparts a very excellent degree of elasticity to the finish, and minimises the liability of the finish peeling or powdering off when the leather is subjected to strain, and at the same time increases the fastness to rubbing and rain-resisting properties. Unfortunately, rubber latex, both on exposure to light and to heat, is liable under certain conditions, which at present have not been investigated, to impart in a very short period of exposure to light an opaqueness to the exposed portions which gives an appearance of a very serious fading. This difficulty, whilst perhaps not insurmountable, is one which renders the employment of latex very unsuitable.

#### Rubbing Troubles

Considerable trouble has been experienced by the leather manufacturer in the application of pigment finishes to leather in respect of their liability to be easily removed by rubbing. In the earlier days of the application of this particular type of finish many examples were so loose to this test that slight rubbing of the surface with a dry handkerchief resulted in the removal of a very material proportion of the pigment finish.

Another defect that is still being experienced to some degree is the liability of the pigment to powder and leave the surface of the leather when the latter is handled or subjected to strain. Serious claims have arisen, for instance, in cases where the colour has rubbed off a lady's handbag and stained her dress.

The attainment of a colour fast to wet rubbing has difficulties that have not yet been absolutely surmounted. Whilst some manufacturers have succeeded in obtaining a reasonable degree of fastness on their products, there are many whose finished leather is considerably lacking in this desired property. Part of the trouble that has been experienced can be traced

to the fact that, in consequence of the continued demand for cheap leather, the leather manufacturer has had to supply a finished product that necessitates the application of a thick film of a pigment finish in order to render it more presentable and pleasing to the public eye, instead of relying, as in the period before pigment finishes were used, exclusively upon the dyer's art.

Pigment finishes, whether used with either cellulose as a binding agent or with aqueous solutions of shellac and casein, are extremely difficult to apply. These finishes were originally intended to be a mere top dressing with a view to giving the desired degree of uniformity of colour to the finished article, but they are now in many instances employed in the form of a thick film as colouring agents.

#### The Use of Cellulose

The present day demand is for a colour which is fast to wet rubbing. This requirement is extremely difficult to meet, and is practically only possible by the use of cellulose, although reasonable fastness is obtainable by the insolubilising of gelatine or fish glue, used as a binding agent, by formaldehyde. The writer's personal experience is that cellulose finishes are chiefly suitable for application to leathers of a non-stretchable type, such as in the finishing of skivers and moroccos. This class of finish is unsuitable for shoe leather and clothing leathers finished on the grain side. There is no gainsaying that where a colour absolutely fast to rubbing is demanded, cellulose offers advantages over the water pigment finishes. What is wanted in the case of the latter is some binding agent, still to be discovered, that will form, when dry, a reasonably elastic film that can be rendered insoluble by treatment with some chemical agent that possesses no deleterious influence upon the leather. There is no doubt that there is a very considerable scope for research in this direction, and the requirement is a pressing one.

A considerable degree of success has been met with in respect of fastness of colour to wet rubbing in the case of hat leather. It is now well known that these goods are dyed in such a manner that the colour is quite fast to washing, and an insoluble finish consisting of isinglass is then applied while the leather is in the wet condition; this process produces a leather the colour of which can only be removed with very great difficulty by the usual rubbing test. The method so employed might in many cases be applied with advantage to other leathers.

#### Fastness to Perspiration

In the case of leather used for wearing apparel, fastness of colour to perspiration, although not at present demanded, will be wanted sooner or later. Many dyestuffs quite fast to light and rubbing are removed by perspiration. The chemical character of perspiration is dependent upon the health of the wearer, and may be either acid or alkaline. A test which has been suggested for obtaining data as to whether a particular dyestuff is fast to perspiration or not is to roll a piece of the leather to be tested in pieces of white cotton and woollen material, previously soaked in a 10 per cent. solution of common salt to which a few drops of ammonium acetate have been added. This is placed in a test tube, tightly corked and kept in an oven at a temperature of 40° C. for 24 hours.

A well-known leather manufacturing firm used to test dyestuffs for perspiration by wrapping a piece of leather in a white textile material and placing it under the saddle of a draught-horse for one or two days so as to become thoroughly moistened with perspiration. Tests made on leather in this way are by no means entirely conclusive, but are only a test for alkaline perspiration, as the perspiration of horses is almost universally of an alkaline character. Perhaps a better method is to enclose a piece of leather in white paper and have this worn as socks in the boots of a number of different individuals.

#### Fastness to Washing

The manufacture of white washable leather by the formaldehyde process drew the attention of glove leather buyers and glove manufacturers to the possibilities of leather that could be washed and thereby regain its original new appearance. A demand has in consequence arisen for coloured leather which can be subjected to the same treatment without material loss or change of colour. Washable glove leather has attained a certain amount of success, and following upon this success the thoughts of other leather users have been directed to demand a leather that would stand being washed with soapy

water without the colour being affected. This requirement has even been extended to a demand for shoe leather which will stand washing.

The colouring of leather so as to withstand the drastic treatment of washing in strong soap solutions, often of an alkaline character, is by no means easy. In the dyeing of finished grain leathers to be subjected to this treatment special selection of dyes must be made that have been tested and will withstand the treatment, and then the leather must be treated with a pigment finish of an absolutely insoluble character, unaffected by a weak alkali. This latter requirement practically limits the finish to the employment of a cellulose solution. Degraded, velvet and suede leathers should be treated after dyeing with a solution of some suitable tannin extract, and the colour fixed by the addition of sodium bichromate, sodium chromate, or copper sulphate. It is, of course, desirable, in order to prevent any liability of damage to the leather in the course of washing in too warm a solution, that the leather should be retanned with basic chromium sulphate to a degree that will enable it to withstand immersion in water at a temperature of 80° C. without material contraction.

#### Fastness to Heat

Many colours which are otherwise quite satisfactory from the point of view of fastness to light and rubbing are materially affected by the application of heat. This remark applies equally to leathers that have been dyed and pigment finished. In the operation of hot plating of leather in the final finishing processes, considerable variation of colour can be effected. Fortunately this is only experienced by the manufacturer himself and does not subsequently arise in the form of a complaint by the customer; and it is easily remedied without liability of loss of reputation by the substitution of a more suitable dyestuff.

In conclusion, the writer would like to strike a personal note and say that he has been very much concerned by the trend of events in the leather dyeing industry during the last

few years, in connection with the requirements demanded of leather manufacturers by their customers respecting various types of fastness. A considerable amount of work has been done at the Leathersellers' Technical College with a view to finding new methods and thus being of assistance to the trade in enabling them to meet the requirements of the user. It would appear that the time is opportune for a research scholarship to be founded at one or other of the technical institutions, for the purpose of carrying out research work with a view to effecting improvements upon modern methods of dyeing and finishing. The writer is of opinion that it cannot be truthfully said that any leather manufacturer throughout the world is at present satisfied with his methods. By founding a scholarship, which would allow some individual to devote his whole time to this important subject, very considerable improvement over present-day methods would no doubt result.

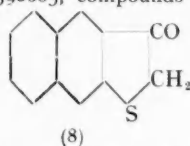
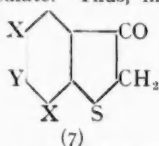
The following are suggestions for the improvement of the production of fast shades of dyed leather:—

- (1) Greater care should be taken in the selection of dyestuffs.
- (2) In the purchase of dyestuffs it is reasonable to demand from the dye manufacturer a guarantee of fastness according to one or other of the standards that have been set up as a criterion by the various research associations functioning in the textile industries.
- (3) Standardisation of the coloured leathers made by the various leather manufacturers should be put into effect, limiting the number of shades of dyed leather to those which can be guaranteed as reasonably fast.
- (4) Every leather manufacturer, before putting on the market any leather which has been finished by the use of new materials, should subject this to the most searching tests. A number of disastrous experiences with cellulose finishes and their subsequent behaviour on storage are recent examples of the necessity of taking careful precautions in this direction.

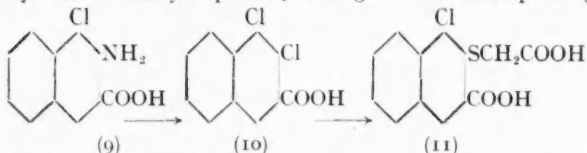
## Basic Intermediates for Dyestuffs: No. XXX.—Some Thionaphthene Derivatives (Continued)

By "Consultant"

VARIETY in shades in the thionaphthene dyes is often brought about by modifications of the aromatic ring either by substitution or by fusion with other rings. In some cases, substitution can be brought about by action on the parent dyestuff, as, for example, in the sulphonation and halogenation of thioindigo, but more frequently the presence of the necessary substituent or fused ring must be brought about by the use of a suitable intermediate. Thus, in U.S.P. 1590685, compounds of the

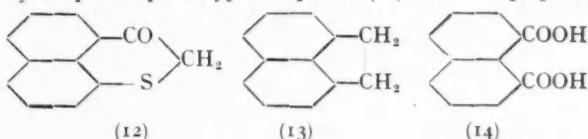


formula (7) are obtained by the halogenation of a thionaphthene substituted in the position Y by a halogen or alkyl group. More interesting intermediates are those derived from the naphthoxythiophene group (8), more especially those produced from the corresponding 1-chloroderivative, which give rise to dyes which are very light- and chlorine-fast. The basic intermediate for this latter compound is naphthalene-1-chloro-2-amino-3-carboxylic acid (9), which is treated by the Sandmeyer process, and gives the corresponding

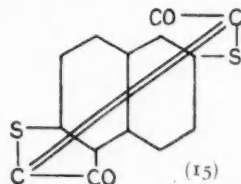


dichloro acid, which, when condensed with thioglycolic acid condenses through the  $\beta$ -chlorine atom only, giving the substituted thioglycolic acid (11). The treatment of this follows exactly that described under thionaphthene itself in the previous article (see THE CHEMICAL AGE, August 10, 1929, Dyestuffs Supp., p. 12).

From naphthalic acid, it is possible to obtain the *peri*-naphthoxypheniophenes, which are capable of condensation with the usual sulphur or nitrogen intermediates to give indigoid dyes. *peri*-Naphthoxypheniophene (12) can be prepared



from acenaphthene (13) by oxidation to naphthalic acid (14). This compound can be converted to naphthalimide and thence to aminonaphthoic acid and naphthoxypheniophene in the same way as phthalic acid is converted through anthranilic acid to thioindoxyl. An interesting example of the application of the thionaphthene principle is the formation of the green vat dye (15) (cf. D.R.P. 380712) by the internal condensation



of 6:5:2:1-naphtho-bis-ketodihydrothiophene in the presence of chlorosulphonic acid. It is supposed that this compound is an internal thioindigo.

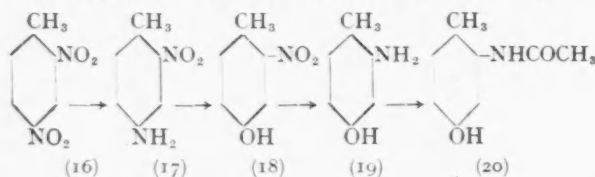
#### Methoxythionaphthene

In order to illustrate the general methods available for the production of substituted thionaphthenes, the technical production of methoxythionaphthene will be described. The first step is the preparation of 2:4-dinitrotoluene, which may be obtained by the nitration of toluene as previously described.

Sulphuric acid of 95 per cent. strength (7 cwt.) is mixed with 75 per cent. nitric acid ( $4\frac{1}{2}$  cwt.) in a nitrating pan fitted with cooling and stirring gear, and toluene (4 cwt.) is run in at such a rate that the temperature does not exceed  $60^{\circ}\text{C}$ . The stirring is continued for 24 hours, during which mononitration proceeds to completion.

After this period, the acid layer is drawn off, and fresh acid ( $13\frac{1}{2}$  cwt. of 95 per cent. sulphuric;  $4\frac{1}{2}$  cwt. of 78 per cent. nitric) is added, and the mixture warmed at  $100^{\circ}\text{C}$ . until dinitration is complete as indicated by the setting point of a sample. The process can be more expeditiously carried out at a temperature of  $115^{\circ}\text{C}$ . The oily layer is allowed to settle and run into cold water, when the DNT solidifies; stirring is maintained in the wash water during solidification in order to granulate the product. The dinitrotoluene is ready for use after a second melting and granulation under water.

Conversion of the dinitrotoluene to *o*-nitro-*p*-toluidine is effected by sodium sulphide reduction. The dinitro compound

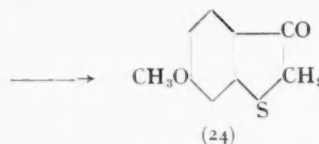
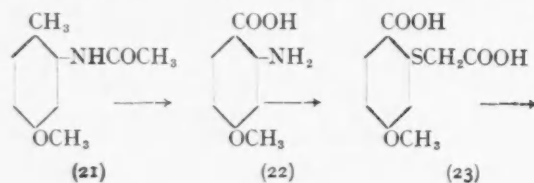


(16) (1 cwt.) is melted and rapidly stirred to an emulsion with boiling water (400 gal.). When a sufficiently fine emulsion has been formed, the reducing solution (48 lb. of sulphur dissolved in  $1\frac{1}{2}$  cwt. of sodium sulphide crystals and 60 gal. of water) is run in gradually. The heat given out by the reduction is enough to keep the batch gently boiling, but when all has been added, the batch is boiled for three hours by indirect steam. Reduction is complete when a filtered sample yields no crystals on the addition of hydrochloric acid. At this point the mixture is filtered hot and allowed to crystallise in tubs, the crystals of nitrotoluidine being removed after standing. The dried crystals are milled to a very fine powder before conversion to nitrocresol by diazotisation.

#### Nitro- and Amino-Cresol

The *o*-nitro-*p*-toluidine (90 lb.) is dissolved in dilute sulphuric acid ( $1\frac{1}{4}$  cwt. 95 per cent. acid in 90 gallons of water) and diazotised by the addition of sodium nitrite (70 lb.). The conversion to the nitrocresol (18) takes place when the diazo solution is gradually warmed, and a short heating with steam suffices to complete the transformation; the solution may be made alkaline with soda ash, and the nitro compound reduced straightway to the amino-cresol (19) by means of hydrosulphite, without the isolation of the nitro compound. When this is done, the theoretical amount of the hydrosulphite is run into the alkaline solution of the nitro-cresol and the mixture boiled until reduction is complete. The amino-cresol separates when the solution is made neutral with hydrochloric acid. It forms brownish leaflets, m.p.  $144^{\circ}\text{C}$ .

Before the methyl group of *o*-amino-*p*-cresol can be oxidised to the carboxyl group, protection must be afforded to the amino group. This is brought about by the usual process of acetylation. The amino compound is treated with four times its weight of glacial acetic acid containing 0.5 per cent. of sulphuric acid, and the mixture heated under reflux. The reaction takes about ten hours, after which half of the acid is removed by distillation, and the residue poured into water. The amount of water is chosen (about one gallon for each pound of amino acid) so that on cooling the acetylaminocresol (20) separates in almost quantitative yield. After filtration, washing and drying the hydroxyl group is methylated. The acetyl amino compound (165 lb.) is dissolved in 25 per cent.



caustic soda solution (20 gals.) and the solution diluted with water (100 gallons). Methyl sulphate (200 lb.) is run in slowly and the whole agitated in the cold until methylation is complete. This may be taken as the point at which a filtered sample solution gives no precipitate on acidification. The product is isolated by dilution with water and filtration, and when recrystallised, melts at  $96^{\circ}\text{C}$ .

#### 4-Methoxy-2-Aminobenzoic Acid

The oxidation of the methyl group to the carboxyl group can be done with potassium permanganate without affecting either the acetyl amino or methoxy groups. The compound from the last stage (2-acetylaminocresol) is suspended in water at  $80^{\circ}\text{C}$ . and potassium permanganate slowly added. The amount of permanganate used is one-sixth of the weight of substance to be oxidised. The solution is maintained at  $80^{\circ}\text{C}$ . until reduction is finished, and a little permanganate should be reserved so that at the end of reduction there is no more excess of permanganate than will give just a faint pink tinge to the solution. This pink colour is removed by the use of the minimum quantity of bisulphite liquor, the solution is steam-heated to boiling, and filtered from the manganese mud. Hydrochloric acid is then added until the solution is distinctly acid; on cooling, 2-acetylaminobenzoic acid separates in needle crystals, m.p.  $197-198^{\circ}\text{C}$ . Before it can be used for the preparation of a thionaphthene intermediate the amino group must be regenerated by hydrolysis. The dry acid is boiled with rather less than twice its weight of 35 per cent. caustic soda solution, until a sample gives no precipitate on acidification. The free alkali is neutralised with hydrochloric acid and on cooling the aminoanilic acid (22) separates in needle crystals. The conversion of aminoanilic acid to 5-methoxythionaphthalene-2-carboxylic acid (24) is carried out in the manner described in the previous article on "Thionaphthene Derivatives."

## Death of Dyeworks Employee

### Inquest Adjourned

THE Huddersfield Coroner, on Monday, adjourned an inquest on William Walsh (58), of Leeds Road, Huddersfield, a plumber's labourer employed by the British Dyestuffs Corporation, at Huddersfield, who died at the Huddersfield Infirmary after having been taken violently sick while at work.

Alfred Thompson, of Cecil Street, Huddersfield, a lead burner, said that on Thursday last he and Walsh were repairing a topping still in the glycol department. Walsh went into the still to dry out any water that might have been left. He then remained at the manhole, handing witness his tools. Walsh did not wear a respirator when he was outside the still. Walsh complained about feeling sick, and when witness got out of the still into the fresh air he also felt sick. Later he took Walsh to the ambulance room, and later to the Infirmary. In answer to Mr. H. M. Atkinson, a factory inspector, Thompson said that one of the assistant chemists had given a certificate that the still was free from gas and fumes. The lead at the side of the still had become defective, and some of the contents of the still might have lodged behind the lead linings.

J. Bottomley, an assistant chemist, said that the still had contained ethylene chlorhydrin, from which ethylene dichloride was obtained. The fumes from the latter were not considered excessively dangerous. He did not know that the lead lining was defective. If that report had been brought to his notice he would have regarded the still as likely to be dangerous.

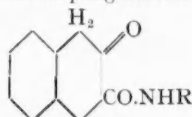
Dr. Guest, pathologist at the Huddersfield Infirmary, said that he was told that the man had inhaled chlorine, and the post-mortem signs were in accordance with death having taken place from chlorine poisoning. He did not know much of the two substances which the still had contained, but similar substances were known to be poisonous.

The Coroner adjourned the inquest for the purpose of obtaining information from the Home Office as to the toxic effects of the two substances mentioned.

# Dyes and Their Application: Recent Technical Progress

By L. J. Hooley

ONE of the difficulties in dyeing colours of the Naphthol AS series has been that the goods padded with the base, e.g.,  $\beta$ -oxynaphthoic arylide, are liable to "fade," if not immediately developed. "Fading" is slightly misleading to those unfamiliar with the process as it is not of course the finished colour which is referred to. The result is that on passing through the diazo-solution, the dyeing comes up weaker. Thus fading takes place much more readily if the padded cloth is dried. Fading is due to the naphthol going over into some form which will not couple. It is sometimes spoken of as decomposition of the sodium salt of the naphthol compound into the free naphthol and caustic soda. It may be an isomeric change to a non-coupling ketonic form, as

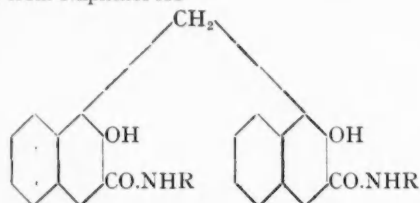


## Prevention of Decomposition

Whatever the cause, the change takes place very readily with acids, the carbon dioxide of the air being sufficient, although in actual practice this is not the only factor. The decomposition can be delayed or prevented either by using a greater quantity of caustic soda in the padding or by increasing the acid character of the naphthols by additional acid groups. The former can only be used to a limited extent, or there is the possibility of salting out and the liability of too much alkali accumulating in the coupling bath, with failure there. The addition of formaldehyde is of especial value and its influence is considered to come under the latter heading. Rath describes the effects with different naphthols under different conditions (*J.S.D.C.*, 44, 10). Higgins discusses the mechanism of this protective action from the theoretical standpoint (*J.S.D.C.*, 43, 213). It is still not clear as to what is the full action of the formaldehyde; however formaldehyde is a very reactive substance capable of forming compounds labile or stable with a variety of groups, and some uncertainty is not surprising.

## The Action of Formaldehyde

One possibility is that the formaldehyde combines with two molecules of the base, as it is already known to do with  $\beta$ -naphthol and  $\beta$ -oxy-naphthoic acid. Kurt Brass and Sommer (*Ber.*, 61, 993) have isolated a similar compound prepared from Naphthol AS



This constitution was determined by acetylation, analysis, molecular weight determination and reduction with cleavage to 1-methyl-2-hydroxy-2-naphthoic acid.

They have also shown that it couples with two molecules of phenyl diazonium chloride to give the same dyestuff as Naphthol AS itself—so that loss of the methylene group must take place. There is a parallel for this in the case of  $\beta$ -dinaphtholmethane.

## Effect of Salt

Turner has investigated the influence of various factors in the dyeing of the naphthols on wool (*J.S.D.C.*, 44, 278). Among these was the addition of common salt to the padding bath. Highly ionisable salts are supposed to protect wool by retarding the ionisation of the strong alkali. Turner confirmed this protecting action in the case of salt. Its use is limited by the fact that it soon causes salting out; with Naphthol AS for instance the highest concentration is 3.5 per cent. and precipitation occurs even with this strength on standing.

As regards the use of alkalis in applying the naphthols to animal fibres, Everest and Wallwork have made a notable advance in finding that if soap is used in dyeing with the

arylides of 2:3-hydroxy-naphthoic acid the affinity is sufficiently great for alkali to be unnecessary, excellent dyeings being obtained. This is applicable both to the arylides of beta-oxy-naphthoic acid and beta-naphthol itself, the latter in fact having better affinity for animal fibres (*J.S.D.C.*, 44, 101). The article contains a very interesting historical summary of the development of the insoluble azo colours. The new processes are covered in British Patents 283,347, 283,838 and 283,839.

Rath also has an article on the application of the Naphthol AS series to animal fibres in the *Textilb.*, 9, 585.

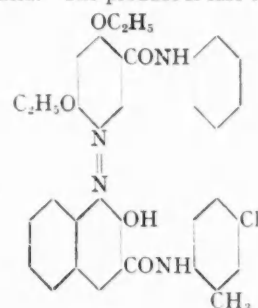
## Synthesis of New Bases

The search for new and heavily substituted anilines as developers for the naphthol bases has brought forward the following additional derivatives:—

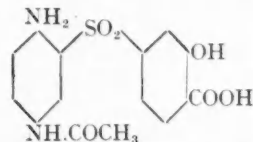
- Benzene-1-amino-3-chlor-2,4-dimethyl.
- " 1-amino-5-chlor-2,4-dimethyl.
- " 1-amino-6-chlor-3,4-dimethyl.
- " 1-amino-2,3-dichlor-4-methyl.
- " 1-amino-2,4-dichlor-3-methyl.
- " 1-amino-2,5-dichlor-4-methyl.
- " 1-amino-4,5-dichlor-3-methyl.
- " 1-amino-4,6-dichlor-3-methyl.
- " 1-amino-3,4-dichlor-2,5-dimethyl.
- " 1-amino-3,5-dichlor-2,4-dimethyl.
- " 1-amino-4,6-dichlor-2,5-dimethyl.

these being described in the following patents: 287,749, 278,761, 287,908, 296,473 (I.G.F.A.-G.).

Alkyl-, aralkyl-, and aryloxy-derivatives are also becoming of importance. Thus in U.S.P. 1,671,442, 2,3-hydroxy-naphthoic arylamides are coupled with a diazotised arylamine-amino-naphthol-methyl-ether, violet shades being thus obtained. Similar products are described in B.P. 294,291, while in 294,883 (S.C.I.B.) diazo compounds of aryl- or aralkyl-ethers of *o*-aminophenols are coupled with anilides of 2,3 oxy-naphthoic acid. In 297,362 (I.G.F.A.-G.), somewhat similar compounds from 3-amino-benzoic acid are used, while in the 4-position there is also a hydroxy group similarly substituted, with also a similar group in the 6-position or else chlorine. The products are bordeaux in shade. In an example, a vivid bluish red is obtained from the 5-chlor-2-toluidide of 2,3-oxy-naphthoic acid and the diazotised anilide of 3-amino-4,6-diethoxy benzoic acid. The product is fast to chlorine.



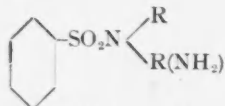
Among products of the sulphone and sulphonamide type there are in B.P. 288,788 (I.G.F.A.-G.) 2,5-diamine-dichlor-sulphones



with acetyl or alkyl groups in the 5-position as well as oxy and carboxylic acid groups in the benzene nucleus, which does not contain an amine. These are diazotised and used for producing monazo and polyazo dyes including developments dyes with Naphthol AS bases.

Other sulphones are described in 255,072 (I.G.F.A.-G.) and

in 279,146 (I.G.F.A.-G.). In the former sulphonamides of the general formula



are coupled with oxy-naphthoic arylides. Thus the diazotised *o*-amino-phenyl-methyl-amide of benzene sulphonic acid and 2,3-oxy-naphthoic acid-*o*-toluidide give a scarlet red, 2-amino-anisole-4-phenyl-sulphone is an example of the type to be found in the latter patent.

In 281,795 (B.D.C., Payman, J. B., and Wignall, G. H.) the preparation of sulphonamidoarylates of 2,3-oxy-naphthoic acid is described. These are obtained by condensing an oxy-

naphthoic acid with aromatic sulphonamides containing primary or secondary amino groups as substituents, for example, 2,3-oxy-naphthoic acid is condensed with aniline *m*-sulphonamide using phosphorus trichloride.

Among miscellaneous products are those of B.P. 275,174 (Rohner), where arylamides of 2,3-hydroxy-naphthoic acid are coupled with tetrazo derivatives of diaminoazo compounds obtained by coupling diazotised mono-oxalyl derivatives of benzene with the middle components and then removing the oxalyl group by hydrolysis. In this way blacks may be obtained.

Further sulphonation products are described in B.P. 291,965 (I.G.F.A.-G.), where 6-hydroxy-2-naphthoic acid is treated with concentrated sulphuric acid at 50–80°C. A mixture of the 4, 7, and 4:7-sulphonic acids is obtained.

## Dyestuffs Markets: The Month's Business in Review

### From Our Own Correspondents

#### Lancashire

NOT since the black days of 1926 have the streets of Manchester been so empty of lorries—the few that pass are only half filled. The shortage of yarn and cloth is still very marked; the legacy of the cotton stoppage accentuated by the holiday period.

Future prospects are not made any more rosy by the publication of a book by Mr. Arno S. Pearse, the secretary of the International Federation of Master Cotton Spinners and Manufacturers Association. He has been investigating the cotton industry of Japan and China, and shows how it is that they have been able to capture such a considerable portion of our Eastern markets. Although mills in Japan are very much more costly per spindle than in Lancashire, the higher overhead charges are met not only by a wage scale less than half of that obtaining here, but also by running the machinery for 17 hours per day in two shifts, instead of the eight hours (or less) worked here. Many weaving sheds are equipped with the Toyada automatic looms, enabling one operative to look after 50 looms; and the average number of ordinary looms tended by one weaver is 5.5, as against 4 in this country. The net result is that Japan, with 6,250,000 spindles, in weight of cotton consumed has beaten England, with her 57,000,000 spindles.

It is in the bulk trade of cheap cloth that Lancashire is suffering so heavily. This is reflected clearly in the trend of the sale of dyestuffs. There is practically no demand for the common direct and basic dyes. But fast to light colours are being called for more and more, whilst the sales of "Naphthols" and vat dyes are quite satisfactory. There are being used largely for yarns to be woven into cloths to be subsequently bleached.

The artificial silk trade does not appear to have been affected by the general depression, and here, too, the faster dyestuffs are growing in favour. Now that it has been shown to be practicable to dye both wool and silk with the fast azoic colours, a further considerable increase is anticipated in the consumption of these bodies, especially since the range of available shades has been rounded off by the introduction of Naphthol Violet and Fast Violet B base, though it is not probable that on cotton these will take the place of such vat dyes as Caledon Brilliant Violet or Purple 2R. Indigo still remains very quiet, and there is very little demand for fast dyes for printing.

#### Scotland

BUSINESS in Scotland during August has been fairly steady. Fortunately the cotton stoppage was brought to an end before the effects were beginning to be felt seriously here. Practically all the holidays are over now, and there seems no reason why a run of several months of good steady business should not be expected.

In the wool industry, matters are still not quite as satisfactory as is desired. Buyers are reported to be holding off for lower prices in view of the falling prices of wool, while manufacturers state they cannot afford to make any further reductions. Work is being put on hand now for patterns

for the 1930–31 winter season. Woollen dyers remain only moderately busy. Round about the Glasgow and Barrhead districts, dyers, printers and finishers have a fair amount of work on hand, but the position is less satisfactory in other quarters.

The United Turkey Red Co. have declared a 3 per cent. interim dividend, which is the same as last year. At the first general meeting of the North British Artificial Silk Co., a report of progress was given. Production was first reached at the beginning of August, when a saleable silk was turned out. It is planned to reach full production on a gradually rising scale by the end of this year.

#### Italian Exports of Dyes, Colours and Pigments

ITALIAN exports of dyestuffs, pigments and colours have increased considerably since 1913. In that year the total exports of all these classes of goods amounted to some 7,000 metric tons, as compared to an average of nearly 10,000 tons in the three years 1925–1927. Moreover, the growth of exports has been accompanied by a marked decline in imports, which have fallen from a total of 18,000 tons in 1913 to an annual average of some 9,000 tons in the three years 1925–1927, a figure below that for average exports. On the other hand, the steady growth of Italian productive activities, accompanied by the growing requirements of the home market, has greatly increased the consumption in Italy. These three considerations point to the rapid growth of the dyestuff and colour industry, which is not only freeing Italy from its dependence on foreign supplies, but has given rise to a considerable export trade. The largest increase has been in the production of dyestuffs, of which in 1913 barely 50 tons were exported, as compared with an average of 2,500 tons in 1925–1927; while imports have declined from 7,000 to less than 1,200 tons in the same period. In minium and litharge, exports have risen from 100 tons in 1913 to an average of 2,500 tons in 1925–1927, while imports have fallen from 520 to 100 tons a year; in unspecified colours, exports have risen from 850 to 1,000 tons a year, while imports have fallen from 2,000 to 600 tons; in paints, exports have risen from 270 to an annual average of 500 tons, while the decline in imports has been very marked, 1,200 tons instead of 3,000.

#### Centenary of Schimmel and Co.

ON September 1 the famous German firm of Schimmel and Co., of Miltitz, near Leipzig, manufacturers of essential oils, perfumes, etc., reached the hundredth anniversary of its foundation. Among the eminent chemists who have been employed by the firm are Julius Bertram (1878–1900), C. von Rechenberg (1883–1917), E. Gildemeister (author of the well-known book *Die ätherischen Öle*) and Heinrich Walbaum. At present, there are engaged at Miltitz 23 chemists and engineers, 150 commercial and technical employees and 278 operatives.





# Dyestuffs Monthly Supplement

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## A British Colour Council

ON Wednesday, at a largely attended meeting in the Connaught Rooms, London, presided over by Lord Ebury, it was unanimously decided to establish a British Colour Council, and a representative provisional committee was appointed to undertake the organisation. The full text of the resolution explains the objects in view perhaps better than any explanatory statements:—

"That this meeting hereby agrees to the establishment of an organisation the title of which shall be 'The British Colour Council,' the objects of which shall be the determination, co-ordination and propagation of colour tendencies, and further agrees that a provisional committee be appointed who shall be authorised to carry through all preliminary arrangements, including constitution and rules, finance, staff, premises, and such other matters as the provisional committee may consider necessary; a report to be furnished to a further meeting."

## A Representative Attendance

The meeting was very largely composed of representatives of the textile industries—Yorkshire and Lancashire being apparently the predominating influence. The speakers represented woollen, cotton, lace, and shoe manufacturers, dyeing organisations, retail distributors, and fashion and design experts. They included Mr. J. Sharp (director of the Bradford Dyers' Association), Mr. E. H. Symonds (Reville, Ltd.), Mr. E. T. Walker (Wolsey, Ltd.), Mr. Forest Hewit (Calico Printers' Association), Mr. R. W. Guild (Bradford and District Manufacturers), Mr. F. H. Briggs (curriers, tanners and dressers), Mr. J. Kerr McCallum (British Cotton and Wool Dyers' Association), and Sir Ernest Jardine (Nottingham Chamber of Commerce). One could not avoid noticing the predominance of the purely commercial interests, and the absence of the technical and scientific sides of the colour industry. There were, however, present a small but authoritative group of the latter class, including Mr. C. Le Maistre, director of the British Engineering Standards Association; Dr. H. H. Morgan, chairman of the Colour Standardisation Committee of the B.E.S.A., and a former president of the Oil and Colour Chemists' Association; Mr. Graham, secretary of the Paint and Varnish Manufacturers' Federation; Dr. New, representing the Paint and Varnish Research Association Laboratory at Teddington; and Mr. Whitaker, of Courtaulds, a well-known authority on dyeing and dyestuffs. It was not until a late stage of the proceedings that the technical point of view was introduced, and this was done so well by Mr. Whitaker, Mr. C. Le Maistre, and Dr. Morgan that Lord Ebury at once welcomed their contributions to the discussions and expressed the satisfaction of the meeting at the prospect of technical and scientific interests being adequately represented.

## Colour Chaos

It did not need much advocacy to convince the meeting of the need of somebody to exercise advisory and directional influence over what are called colour tendencies. The growth of the colour sense, not only among educated and artistic people but among the mass of the people, has been enormous in recent years, and the people who have to provide for its satisfaction—manufacturers, dyers, and

dyestuff manufacturers—have no sort of barometer as to the colour vogues and fashions that will predominate in a season six or twelve months ahead. No central body can, of course, impose its will on the trade or on the popular public fancy, but it may, by consultation among all the relevant interests, indicate the general trend of fashion in colour and indirectly do a great deal to give the authority of fashion to the colours selected. Admittedly the Lyons and Basle colour cards do much to suggest the forthcoming fashion tones and to bring them into general use, and the American colour cards, backed by the enthusiasm of Mrs. Mary Rorke, are said to be a predominant influence on the colours used in that land of slogans. The quotation of the American example, however, may be overdone. There business is run largely on slogans, which the public obey in an almost regimental fashion, whereas here there is the obstacle of a highly individualist temperament to be reckoned with. That, however, is no reason for complete inaction. Admittedly, a British colour card prepared by a representative committee of experts would be a valuable guide to all engaged in meeting the demand for coloured productions of every kind, and its general adoption would still leave room for individualist taste. Emphasis was placed on the importance of its early issue.

## Standardisation

Another trouble at present is the confusing variety of names employed to describe the same colours—Saxe Blue was mentioned more than once as an example. Here what is wanted is some system of standardisation by which the names or numbers of colours will stand for some definite and recognised tone. On this point the meeting heard with interest Dr. Houlston Morgan's statement that a standardisation committee for the paint and varnish industry had been at work for about three years on this problem of settling standards for certain colours, and his announcement that within the next month or so they hoped to produce a British colour card for the use of the industry. It was a very useful reminder to the conference that fashion and advertising experts are not the only authorities on the subject of colour, and that the new British Colour Council will have, like other bodies, to bring in the scientist—as, indeed, Lord Ebury had suggested at an early stage of the meeting—if the work of the council is to be on sure foundations. Mr. Le Maistre also added some useful remarks on the work of the Engineering Standards Association, and it was notable how quickly the conference recognised that here was an essential aspect of the question which the purely commercial interests had almost entirely overlooked. It was curious that throughout the discussion no single reference was made to that monumental work the *British Colour Index*. One was left wondering whether our expert business men and fashion experts had ever heard of it.

## Technical Aspects

Another pointed and much appreciated contribution to the discussion from the technical side came from Mr. Whitaker, of Courtaulds, who testified to the innocence of customers who demanded the exact reproduction of colour patterns on the assumption that a given colour could be indiscriminately applied to such different fabrics as

wool, cotton, silk, artificial silk, and mixtures of two or more of them. The selective action of certain fabrics on certain classes of dyes—a commonplace among dyestuff makers and users—seemed to be quite a new idea to the shop-keeping interests, and the difficulty of maintaining exactly the same shade over lots of small batches also seemed to impress them. Very dryly Mr. Whitaker announced that he never undertook to dye two shades exactly alike. The matter was further complicated now, Mr. Whitaker pointed out, by the demand of the drapers that "fadeless" should be strictly interpreted in a literal sense, though that is not quite the sense in which the word is used among dyestuff authorities, and Mr. Whitaker quite carried the meeting with him in his plea that the Council must include technical men who understood dyestuffs manufacture and application scientifically and who were capable of appreciating the limitations within which shades were expected to be given to goods differing widely in fibre. Mr. Whitaker was himself added to the provisional committee, and a suggestion by Dr. Morgan that the paint and varnish authorities would also be glad to nominate a representative was at once cordially accepted by the Chairman.

### A Good Start

The conference, as a first step, may be described as quite a success. It produced a general impression of the great importance of a more thorough study of the colour problem in relation to the textile and allied trades, and it gave the purely shop-keeping end a new vision of the part that science and technology must play in this as in almost every other matter. It may be added that, at the opening, Lord Ebury read communications warmly approving the objects of the conference from Imperial Chemical Industries, Ltd., Courtauld's, the Colour Users' Association, the Calico Printers' Association, the Chemical and Dyestuffs Traders' Association, and several other bodies.

After the conference Dr. Morgan expressed the opinion that, while the discussion had included much of a rather hazy and general character, it had served a good purpose. The first object, the issue of a carefully considered colour card well in advance of the season when the colours would come into use, must be of considerable advantage to all concerned in the colour industry. The Council, he emphasised, must also address itself to the standardisation problem, not, as some seemed to think, with the object of forcing certain colours on the public, but in order to establish recognised standards by which the leading colours could be identified by names or numbers. He thought the movement would gain considerably from the research work in this field that had already been undertaken for the paint and varnish industry.

### Optically Active Dyes

Ingersoll and Adams (*J. Amer. Chem. Soc.*, 1922, 44, p. 2930) have prepared two pairs of optically active dyes derived from *d*- and *l*-phenylaminoacetic acid, one pair of which showed evidence of selective absorption by wool, while the other pair was absorbed in equal amounts. From these facts, they concluded that the process of dyeing is sometimes *chemical* (as evidenced by the selective absorption of the optically active dyes) and sometimes *physical*, because of the absence of selective absorption. Recently Porter and Ihrig (*J. Amer. Chem. Soc.*, 1923, 45, p. 1990) prepared a pair of optically active dyes derived from mandelic acid, the *d*-form of which was selectively absorbed by wool from the racemic mixture, leaving the unabsorbed portion of the dye bath composed of almost a pure *l*-compound.

All of the optically active dyes which have been previously prepared, with the possible exception of "the camphorines" of Sing, Rai and Lal (*J. Chem. Soc.*, 1922,

121, p. 1421) were monoazo dyes whose optical activity depended on one asymmetric carbon atom. If one could prepare optically active tetrazo dyes whose optical activity depended upon two asymmetric carbon atoms instead of one, the evidence for selective absorption, if there were any, would be more pronounced than would be the case with monoazo dyes.

### Dyes with Two Optically Active Centres

From this standpoint, an attempt has been made by N. Uyei to prepare optically active dyes by tetrazotizing benzidine and coupling with an active intermediate. The experiments are described in a thesis entitled "Optically Active Dyes" just published by the author from the University of Illinois. The intermediate tried was *l*-phenyl-(*p*-aminobenzoylamino)acetic acid. The dye thus obtained was extremely unstable and could not be purified. A few attempts to prepare other optically active benzidine dyes in pure form were also unsuccessful. The failure in preparing benzidine dyes in pure form turned the attention to the preparation of other modifications of monoazo dyes derived from phenylaminoacetic acid. For this purpose the latter compound was first resolved into *d*- and *l*-forms. Each form was condensed with *o*-nitrobenzoyl chloride, the nitro group was reduced to an amino group, diazotized and coupled with common intermediates to be converted into a dye.

Of three intermediates tried, beta-naphthol was found to give dyes that are capable of being purified. Thus, not only the racemic modifications, but a pair of enantiomorphs of the dye were prepared in pure form. The rate of absorption of these optically active dyes on wool and silk was studied by colorimetric methods by optical measurements, and by gravimetrically weighing the dyed fibre. No indication of selective absorption was revealed by the use of any of these methods.

### Dyestuff Licences for September

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during September, has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—

The total number of applications received during the month was 612, of which 545 were from merchants or importers. To these should be added 26 cases outstanding on August 31, making a total for the month of 638. These were dealt with as follows: Granted, 564 (of which 516 were dealt with within seven days of receipt); referred to British makers of similar products, 56 (of which 44 were dealt with within seven days of receipt); outstanding on September 30, 18. Of the total of 638 applications received, 560, or 88 per cent., were dealt with within seven days of receipt.

### American Winter Colour Fashions

A special winter card showing eighteen shades has just been issued to members of the U.S.A. Textile Colour Card Association, by Mrs. Margaret Hayden Rorke, managing director. The card portrays the colour high-lights of the recent Paris openings, as well as other outstanding colour developments for the 1929-30 winter season.

"The innovation this year of a special summer card was so favourably received," Mrs. Rorke states, "that the Association has decided to issue these supplementary cards in addition to its regular seasonal colour cards. The Winter Card, which is designed to serve all branches of the fashion industries, in no way lessens the importance of the regular 1929 Fall Season cards, but rather confirms important colour tendencies already forecasted by the Association, and presents, in addition, certain new colour themes for the mid-season."

## Food Colouring Practice in Canada and the United States

By L. Whinyates

*In the following paper, presented at the Twelfth Dominion Chemical Convention, held at Toronto recently, Mr. L. Whinyates (of the Dye and Chemical Co., Ltd., Kingston, Ontario), deals with present practices, dyes used in Canada and the United States, and the need for world standardisation.*

HISTORY does not appear to record the time when foodstuffs were first coloured, but a glance around the shelves of any grocer's or chain-store will quickly show how prevalent the practice has become at the present time. Immediately the question is raised: "Why?" Naturally, one associates colour with flavour, and one's first thought is to consider the colouring of foodstuffs as a little harmless deception played on the general public. Still, we prefer our food coloured, and possibly realise that the pleasing colour of a foodstuff, transmitted through the eye to the brain, is further transmitted to the stomach, stimulating the gastric juices and promoting appetite and digestion. Food colours, therefore, are useful as well as ornamental.

Originally, vegetable colours (including cochineal, of animal origin, and one or two mineral colours) were used for colouring foods, but with the advent of the synthetic colours, the former have now practically fallen into disuse, except in one or two cases. Probably well over 95 per cent. of the colour used in foodstuffs at the present day is synthetic, and it is with synthetic colour that this paper will deal.

### Three Main Factors

The colouring of foodstuffs is governed by three main conditions: (1) The laws of the district in which the products are consumed; (2) The colours must be harmless both to the user and the consumer; and (3) They must be stable in the material in which they are used.

Conditions (1) and (2) are practically the same, as the laws regulating the use of colours in food all have the same objective, namely, the protection of the public against the use of harmful colours, or harmless colours containing harmful impurities. Unfortunately, all countries do not agree on the methods employed in attaining this end. The simplest regulations, that is, those most easy of interpretation, are those which prohibit the use of synthetic colours, but the Argentine is the only important country now in this class. Great Britain gives a list of five harmful colours, but these are merely examples, as the British regulations state that only colours of a harmless nature may be used. These regulations are the most difficult to interpret, but they do cover harmful impurities, which are limited to 1.4 parts of arsenic, or 20 parts of lead, copper, tin and zinc per million.

Whilst on the subject of harmful colours, and also of arsenic as an impurity, it may be mentioned that amongst the general public the idea still persists that all green candies, etc., are coloured with arsenic, and also that all "aniline" colours are poisonous, or at least, not entirely safe for human consumption. There is no idea in the public mind as to the amounts of colour used in foods, and the writer well remembers the gasp of amazement which greeted a statement at a certain Home and School Club, that a 2-gram tube of strawberry colour (samples of which had been distributed to the club members) would colour ten gallons of ice cream, this representing approximately one part of colour in 25,000 of cream.

### Simple U.S. Regulations

The United States regulations for food colours are the most simple of interpretation, because they are the most stringent regulations now in force. A permitted list of colours is given, together with complete specifications, covering tolerances for moisture, insoluble matter, sodium salts, arsenic, heavy metals, subsidiary dyes, etc., and official methods of analysis. The present permitted list includes fourteen colours, two of which have been added during the past twelve months.

Canada, together with Australia and New Zealand, has based its regulations on those of the United States. Canada has a list of twelve permitted colours, of which ten are in the United States list, and one (Butter Yellow) is practically obsolete. This permitted list is as follows:

Reds—Amaranth, Ponceau 3R, Erythrosine.  
Orange—Orange I.

Yellows—Naphthol Yellow S, Tartrazine (water soluble), Sudan I, Butter Yellow, Yellow AB, Yellow OB (oil soluble).

Green—Light Green SF Yellowish.

Blue—Indigo Carmine Disulphonic Acid.

One may remark here that while Naphthol Yellow (Dinitro alphanaphthol) is prohibited in Britain as harmful, the monosulphonic acid (Naphthol Yellow S) is permitted both in Canada and the United States. Actually, it seems that very little of this colour is used, as Tartrazine is used almost universally as a water-soluble yellow.

### Suitability for Purpose

It should be noted here these permitted colours are by no means the only harmless colours, nor, as will be discussed later, are they necessarily the most suitable colours for use in colouring foods. One of the soundest ideas in the making of the original permitted lists was that the permitted colours should be capable of easy identification, and should be easily distinguishable from other colours. The Colour Index of the Society of Dyers and Colourists contains the names of 1,230 dyestuffs, and if a dozen of these were picked at random, and samples submitted to any analyst, he would probably find a long and tedious occupation in identifying each one of the twelve. It is, however, a comparatively easy matter to determine that a colour is not a permitted one. It is not always possible to name a colour, although it may be placed in its particular group, for example, a Rhodamine or Benzopurpurine, but it is quite sufficient to be able to say that a red colour is not Amaranth, Ponceau 3R, or Erythrosine.

The only specification given for food colours in Canada is covered by the following sentence: "The above coal tar dyes must have been manufactured in specifically pure form for food purposes, and must not contain arsenic in excess of ten parts As<sub>2</sub>O<sub>3</sub> per million, reckoned upon the actual weight of colouring matter, nor heavy metals (iron excepted)." (Regulations under the Food and Drugs Act made by Order-in-Council, February 6, 1928.)

### Testing Food Colours

No methods of testing are given in the Dominion regulations, but the U.S. Standards are generally accepted by the Departments responsible for food colour testing. The departments concerned are the Department of Agriculture and the Department of Health, which is responsible for the collection and testing of materials sold and used in the home market. U.S. Standards would apply particularly to such a case as the question of identity of Ponceau 2R or Ponceau 3R. Ponceau 3R is made from pseudo-cumidine, and Ponceau 2R from a mixture of xylydines. In a case of doubtful identity, the colour is decomposed by means of stannous chloride, the amine steam-distilled and extracted, and its boiling range determined. This boiling range, according to U.S. specifications, must be between 220° and 245° C., and I believe these figures are accepted by the Dominion Department of Health.

### Manufacture More Difficult

The manufacture of food colours is a distinctly more difficult proposition than the manufacture of colours, say, for textile dyeing. Care is necessary, not only in the final steps of their manufacture, but also in the manufacture or purification of the intermediates. Many of the intermediates used are commercial in a relatively impure state, containing considerable percentages of isomers, or subsidiary compounds, which will produce isomeric or subsidiary colours in the process of manufacture. This is not usually a serious matter in the case of textile colours, as small variations in shade, due to varying proportions of isomeric colours, can be corrected fairly easily by further additions of colour in the grinding or blending shed. This, of course, is not possible with food colours, nor is it generally possible to remove these isomeric colours once they are produced.

The only solution lies in the use of intermediates of the highest possible purity. Other difficulties in manufacture which may be mentioned are: The iodation of fluorescein to give tetraiodofluorescein or Erythrosine; alkalinity control in the manufacture of Orange I, which acts as an indicator; alkalinity control in the manufacture of Yellow AB and Yellow OB; sulphonation of the benzylethylamine-benzaldehyde condensate to give a trisulphonic acid in manufacture of Light Green SF. In the final case, over-sulphonation gives Guinea Green B, which is permitted in the United States but not in Canada. The result of these difficulties, or perhaps I should say of the overcoming of these difficulties, is that production costs, and consequently prices, are considerably higher than for the usual synthetic colours; when it is realised that under Dominion regulations added colour is limited to 1 part in 3,500 the actual cost of colouring foodstuffs is not very considerable.

#### **Three Classes of Coloured Foodstuffs**

Foodstuffs may be roughly divided into three classes for colouring purposes:

1. Soft drinks, gums, boiled sweets and other transparent goods.
2. Chocolate centres, biscuit fillings, ice cream and similar opaque products.
3. Butter.

The main difficulty in the first class is fading, which may be due to light, sulphur dioxide, high temperature, or occasionally to the action of the raw materials themselves. Generally, the permitted colours are reasonably fast to light with the exception of indigo carmine, which fades appreciably in ordinary daylight. Tartrazine is the fastest to light of the twelve. Sulphur dioxide is largely used as a bleaching agent, particularly for glucose, and interferes very considerably with the colouring of boiled sweets (in which a considerable amount of glucose is used) as a certain amount of sulphur dioxide remains unchanged in the glucose. It is possible to overcome this by using an excess of the colour, but it is generally preferred to destroy the sulphur dioxide by means of cream of tartar before the colour is added.

Amaranth is readily decolourised by sulphur dioxide and also is not particularly stable at high temperatures. In one case, a candy maker was using Amaranth in excess to colour a boiled candy orange, and he was delighted to find that he could use considerably less colour by substituting a mixture of Ponceau and Tartrazine, which stood up to the temperature conditions. A similar mixture was found valuable in colouring orange drinks, where the acid conditions convert the usual orange shade (made by using Orange I) to a yellow. Orange I, of course, cannot be used in alkaline media, as the result is a red; nor can indigo, which is practically bleached by alkali.

#### **Colours Used on an Opaque Base**

When colours are used on an opaque base, similar difficulties apply, increased by the fact that these materials contain very little water, and frequently glycerine is added to retain the full shade of the colour employed. In the case of materials sold dry, such as custard powders, it is practically impossible to give a good colour to the powder without having too much colour present in the final product, after it has been mixed or boiled with water or milk. It is perfectly easy to match either a dry or a wet mixture, but extremely difficult to match both in the same material.

#### **Colouring of Butter**

The colouring of butter provides very little difficulty; the use of a mixture of Yellow AB and Yellow OB in oil solution gives perfectly satisfactory results. One point which must be watched, is the possibility that the colour may be partly thrown out of solution through freezing. If such butter colour is used before it has been warmed, and the dyestuff has not been entirely redissolved, particles of solid colour may be present in the butter, which cause specks in the butter itself on standing.

There are undoubtedly many cases in which oil-soluble colours could be used to advantage in colouring foods, but as there are only two yellows and a yellowish red permitted for use in Canada, these applications are impossible until a larger range of colours is permitted. This, however, is not possible while the Government's attitude is that food colours may be a necessary evil, but still are evil.

In conclusion, it must be obvious that some form of international discussion is desirable, with the object of standardising food colour regulations. As practically every nation is interested in this matter, it should not be extremely difficult to form an international commission with the aid of the various committees, Governmental or scientific, now considering the problems from their own national viewpoints, and societies such as are represented in this convention could be of great assistance in furthering this object.

### **Light as a Research Weapon**

#### **Bureau of Standards Experiments**

THE matching of shades, one of the problems of the modern dyer which have not yet been reduced to an accurate science, may soon be standardised on the basis of data now being collected by the United States Bureau of Standards on the behaviour of light rays in different colours.

Physicists in the laboratory of the Bureau have been making a systematic study of light with relation to shade matching. A spectrograph is taken of dyed samples, and by means of certain prominent lines or bands in the spectrum, the dye of that material is definitely identified and can be exactly duplicated.

In many lines of work at the Bureau of Standards, light is the principal factor used in the test. Some tests are intended to simulate average daylight conditions. For others, only light of a particular colour, or rather wave-length, is used. In many instances only the light or radiation in the invisible portion of the spectrum is used, the ultra-violet and ultra-red. But, because the weather is such an uncertain element, the scientist cannot depend on the sun as a reliable source of energy for his tests. Quick, reliable results, under conditions which can be duplicated, and which are comparable with solar radiation, are necessary.

#### **Spectral Analysis**

A careful spectral analysis was made of the light emitted by the sun and compared with artificial light sources, and for many tests, especially in the study of the fading of fabrics, paper and inks, the carbon-arc lamp is used because its light has been found the closest approach to that of average sunlight. The time of exposure of samples to such light is extended as much as seems necessary. In some cases, as when testing paints and varnishes, a water spray is used to imitate the effect of rainfall. Most of the work mentioned is in the experimental stage, so that just how particular kinds of material will behave remains to be seen.

Everyone knows how difficult it is to obtain an exact colour match for cloth, and it is quite a problem, even for the manufacturer, to duplicate any desired shade. But some interesting work being done by the Bureau may solve the difficulty. Other questions entering into the dyeing of a fabric must be considered, but these should be within control of the manufacturer.

Incandescent lamps are tested to insure that the lamps supplied the Government under contract comply with the specification for candle-power and life. Lamps of an entirely different type are calibrated for manufacturers for use in pyrometers to enable them to keep foundry operations in the working of metals well within the critical temperature ranges required.

By the rotation of a beam of plane-polarized light passing through sugar solution, the percentage of sugar is obtained. By this method the Bureau's sugar laboratory checks the laboratories of the Customs Service, and the duty collected by the Government on imports is based on the results of these tests.

For some kinds of work, monochromatic light, or light dominant in one particular colour, is used, more particularly to ascertain differences in dimension between two objects, or to determine those minute but important changes which occur with change in temperature or when material is in a stressed condition. By interference of light waves, a pattern of light and dark bands is disclosed. The distance from one light or dark band to another light or dark band represents one-half wave-length. This method is used to check the master gauge blocks for manufacturers, the accuracy of the depth of blood-counting chambers used by physicians, dental amalgams, enamels for metal ware, glazes for porcelains, etc.

## Basic Intermediates for Dyestuffs: No. XXXI.—The Chlorobenzenes and Their Derivatives

By "Consultant"

THE introduction of a chlorine group into a dyestuffs molecule often has a very marked effect on the shade of dye obtained, as, for example, in the case of the chlorinated thioindigo dyes. It is very seldom that the introduction of a chlorine atom is done during the last stages of the manufacture of the dye: more often the chlorine is placed in a suitable position in the raw material, and remains unchanged throughout the various processes which follow. Further, the high reactive powers of chlorine when attached to certain aromatic nuclei lend interest to compounds containing chlorine atoms, since they represent potential starting points for the production of new intermediates. An example of this is the compound chlorodinitrobenzene, which will condense through its chlorine atom with aniline to give a dinitro derivative of diphenylamine.

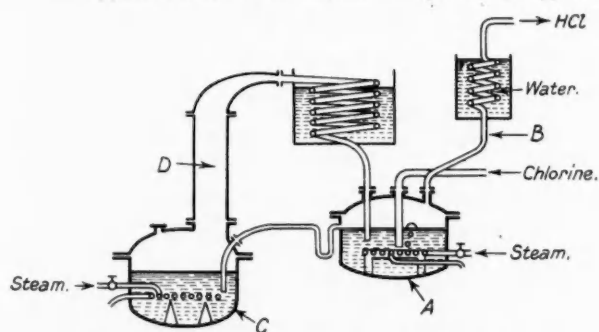
### The Chlorobenzenes

The chlorination of benzene leads to the formation of a mixture of monochlorobenzene with some dichlorobenzene. The process used for such a chlorination will, therefore, depend on the demand for the dichlorobenzene. Where this demand is sufficient to absorb all the production, a very simple chlorination plant will suffice; where, however, there is no demand for dichlorobenzene, it is preferable to use a more complex plant, in which the amount of dichlorobenzene is reduced to a minimum. Aluminium wire is a good catalyst for the chlorination of benzene, but wrought-iron powder is thought by most operators to be equally efficient. With aluminium wire present in the proportion of one part to 1,000 of benzene, an 87 per cent. yield of the mono derivative is obtainable.

The chlorination of benzene has been studied by Bourrion, who has come to the following conclusions:—The amount of monochlorobenzene formed rises as the temperature of chlorination is increased, so that it is advisable to chlorinate at the boiling point in order to increase the yield. At this point the ratio of benzene to monochlorobenzene (measured in molecular proportions) is 8.5 times the ratio of mono to higher chlorinated derivatives.

The normal process for the chlorination of benzene on a large scale is to pass chlorine into boiling, pure dry benzene to which has been added 1 per cent. of dry wrought-iron powder. The chlorine should be completely absorbed, and the progress of the reaction is measured by weighing the amount of chlorine passed in. When this amount corresponds to 35–40 lb. of chlorine for each 78 lb. of benzene the process is stopped and the clear liquid fractionated. It should yield 80 per cent. of monochlorobenzene, 10–15 per cent. of dichlorobenzenes and 5 per cent. of unchanged benzene. When a higher yield of the monochloro-derivative is desired, some process is used in which the chlorinated mixture is made to circulate in such a way that the mono-derivative is constantly being withdrawn from the system, and is not allowed to come into contact with chlorine.

The apparatus shown below is an example of the applica-



PLANT FOR THE CHLORINATION OF BENZENE.

tion of this principle. The chlorination is performed in the cast-iron vessel A, chlorine being led in through one tube and hydrogen chloride passing out through the condenser tube B.

The benzene is mixed with wrought-iron powder as before, and is maintained in a state of brisk ebullition by steam coils. The chlorinated liquid flows continually into the still C, where the benzene is removed from the chlorinated derivatives by means of steam heating. A short column D serves to retain any chlorobenzene which is volatilised. The condensed benzene runs straight into the chlorinating vessel, thus maintaining a continual circulation, the chlorobenzene being removed as fast as formed.

The addition of ferric chloride to the iron used as a catalyst is stated to improve the yield of the monochloro-derivative. In one run, 300 parts of benzene were treated with one part of iron powder and one part of ferric chloride. On distillation of the chlorinated product, 335 parts of pure monochlorobenzene (b.p. 131–3° C.) were obtained, together with 24 parts of pure *p*-dichlorobenzene (m.p. 50° C.) and 13 kg. of a mixture of dichlorinated benzenes. A process has been devised for the continual chlorination of benzene, in which the latter substance is allowed to trickle down a series of layers of quartz pebbles contained in a tower up which a current of chlorine is passing. The ratio of monochlorobenzene to higher derivatives in this apparatus is said to be 100/6, but it is doubtful whether the added complications of the plant involved make this increase in yield of the monoderivative worth while.

### Chlorobenzenes

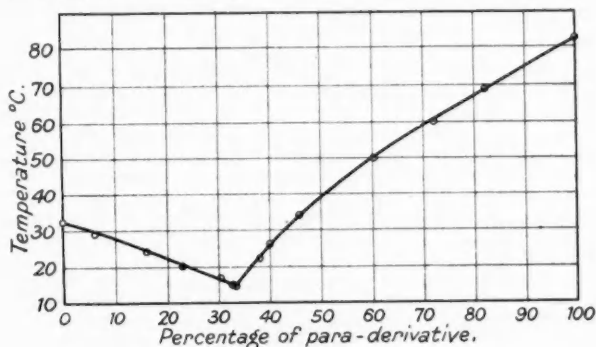
Monochlorobenzene is a colourless limpid liquid, with a pleasant smell. It melts at 45° C. and boils at 132° C., and at 0° C. has a density of 1.128. The dichlorobenzenes have the following characteristics:—

SUBSTANCE.	M.P. °C.	B.P. °C.
<i>o</i> -Dichlorobenzene .....	—	180
<i>m</i> -Dichlorobenzene .....	—	172
<i>p</i> -Dichlorobenzene .....	53	172

It is obvious from the above data that the separation of the ortho- and para-dichlorobenzenes by fractionation is difficult. It is better to cool the mixture of isomers, and allow the para-dichloro derivative to separate out in the solid crystalline form. The residue may then be fractionated, when the ortho derivative will be obtained moderately pure in the later fractions. Paradi-chlorobenzene is of more value commercially than the ortho variety. By the process of nitration and reduction, it can be converted into 2:5-dichloroaniline, a valuable intermediate, and by itself it has been found of particular value as an insecticide, more especially as a preventive from damage by the clothes moth.

### The Nitrochlorobenzenes

When chlorobenzene is nitrated at 0° C., a mixture of the ortho and para isomers is formed, together with a very small amount of the meta compound. Thus, Hollemann found that the proportion of *o*:*m*:*p* was 29.8:0.3:69.9. The actual

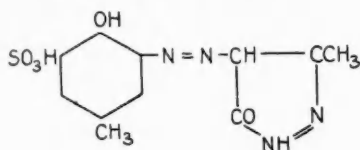


SETTING-POINT OF *o*- AND *p*-DICHLOROBENZENES.

nitration of chlorobenzene is a process which follows in detail the process already described in this series for the production of nitrobenzene, and need not be repeated here. The difficulty



solvents with sufficient chromium compound to give one molecule for each chromable group. The product from 4-nitro-2-aminophenol-6-sulphonic acid and 1-phenyl-3-methyl-5-pyrazolone dyes clear yellowish orange shades, so that the presence of the metal in this case causes, as would be expected, little change in shade. In B.P. 282,783 (S.C.I.B.) shades from orange to red are obtained on animal fibres from an acid bath by dyeing with compounds somewhat similar to those mentioned in the patent immediately above, but obtained from 3-methyl-5-pyrazolone; that using 4-methyl-2-aminophenol-6-sulphonic acid is an orange.



Somewhat similar metal compounds again are instanced as useful for making transparent varnishes in B.P. 293,358 (S.C.I.B.), e.g., the chromium compound of the dye obtained by coupling diazotised 2-aminophenol-4-sulphamide with 1-(3'-sulphamino)phenyl-3-methyl-5-pyrazolone gives orange shades. In B.P. 302,709 (I.G.F.A.-G.), *o*-aminonaphthol monosulphonic acids are used.

#### Sulphur Dyes from Metal Azo Compounds

A new feature is the use of soluble chromium compounds of azo dyes as intermediates for sulphur colours. In B.P. 290,179 (S.C.I.B.), the chromium compound from 2-aminonaphthol-4-sulphonic acid-naphthol is heated with aqueous sodium sulphide and sulphur under reflux to give a dye yielding reddish blue shades from the sodium sulphide vat.

The colours normally regarded as development or ice colours have also been called into service as starting materials in the production of these metal azo compounds. In B.P. 288,983, an arylide of 2:3-oxynaphthoic acid is coupled with a diazotised aminoazo compound and then treated with a metallic salt. Again, in B.P. 300,425, dyes from *o*-oxydazo-compounds and acetoacetic esters or arylides are chosen for treatment with chromium compounds.

#### Mixed Metal Azo Compounds

Interesting mixed metal compounds can be obtained by treating the azo dye not with a metal but with a metal compound of another azo dye; or the metal compound of the same azo dye can be used (B.P. 297,331, S.C.I.B.). In an addition to this patent (B.P. 289,091), similar products are made, but this time they are synthesised on the fibre or in the dyebath instead.

Other particularly complex compounds may be obtained by using a metal azo dye as an intermediate. Thus the chromium compound of the dyestuff from one molecule of 4-chloro-2-aminophenol and one molecule of 5:5'-dihydroxy-2:2'-dinaphthylamine-7:7'-disulphonic acid is treated with copper sulphate and then coupled with diazotised 4-chloro-aminophenol, and afterwards given a second treatment with copper sulphate.

#### Afterchroming Metal Azo Compounds

The use of chromium compounds of azo dyes does not preclude the use of chromium on the fibre as well, if some advantage can be obtained, and examples of this are to be found in B.P. 303,384 (S.C.I.B.). Wool is mordanted with bichromate and tartrate, and then dyed from an acid bath at the boil with the product obtained by chromium treatment of the dyestuff 5-nitro-2-amino-phenol-2-aminonaphthol-6-sulphonic acid; a fast yellowish green is obtained, which stands up better to alkali than if the chroming of the fibre is omitted.

Among other miscellaneous bodies described the following may be mentioned:—

Chromium compounds suitable for cotton are made in B.P. 271,897, by diazotising an *o*-aminophenol or an anthranilic acid and coupling with certain components. As an example, a blue green can be obtained from 4-chloro-5-nitro-2-aminophenol and "J" acid.

B.P. 279,429 (I.G.F.A.-G.), where 1,2-aminonaphthol and 1,8-naphtholsulphonic acid derivatives are coupled and treated with metals.

In B.P. 283,979 (I.G.F.A.-G.), *o*-hydroxy diazo-compounds are coupled with naphthols, and then treated with chromic acid at high temperatures under pressure.

B.P. 294,743: The dye from metanilic acid and salicylic acid is warmed with chromium formate to give a clear greenish yellow.

B.P. 295,594 (S.C.I.B.): Production of metal azo compounds is carried out by treatment in the presence of an alkaline reducing agent with hydrated chromium oxide and a protective colloid. Reducing agents can also be added to the bath.

B.P. 295,944 (S.C.I.B.): Products from 5-nitro-2-aminophenol and *N*-acyld-2-amino-5-naphthol-7-sulphonic acid are used.

B.P. 297,722 (I.G.F.A.-G.): Diazotised 4-chloro-2-aminophenol-6-sulphonic acid coupled with 2:4-dihydroxyquinoline and metallised gives fast bordeaux red shades.

In B.P. 297,478, the metal compounds of azo dyes are nitrosated and reduced. A steel blue dye is obtained in this way using as a basis the dye 1,2-aminonaphthol-4-sulphonic acid- $\alpha$ -naphthol.

## Dye Cartel and Dye Exports

### Five Major Producing Countries

THE formal signing of the sales agreement by representatives of dye manufacturers of Germany, Switzerland, and France was one of the outstanding events of recent international activities. This agreement was signed by three European countries whose export trade in coal-tar dyes in 1927 accounted for more than three-quarters of the quantity of the world's exports and four-fifths of the value. The significance lies, however, in the fact that the signatures gave permanency to an oral agreement which had been in effect for the past three or four years, by enlarging and reducing it to writing. In other words, it was the conclusion of negotiations lasting the past five years.

The participants were the German I. G. Farbenindustrie, the French dye firms Etablissements Kuhlmann and the Société des Matières Colorantes de Saint Denis, and the Swiss companies A.G. für Chemische Industrie (Ciba), Chemische Fabrik vormals Sandoz, which is closely connected with the A.G. für Chemische Industrie, and J. R. Geigy, A.G.

It is understood that the Clayton Aniline Co., the English subsidiary of Ciba, will operate under the terms affecting its parent. Other subsidiaries of Ciba which are said likewise to be affected are the French subsidiary at Saint-Fons, the Polish subsidiary, S. A. Pabianicoise pour l'Industrie Chimique, and the Italian subsidiary, the Sta. Bergamasca per l'Industria Chimica, Seriate.

The agreement, which was brought about by present market conditions, refers exclusively to co-operation in the sale of aniline and other synthetic dyes—especially synthetic indigo. Each country retains its independence as to capitalisation, profits, and commercial development. Much emphasis is laid upon the preservation of independence in technical and commercial details. Though the agreement is referred to as a sales agreement, it is in reality an agreement for regulating and stabilising the prices of the standard dye products.

#### Basis of the Agreement

Authoritative sources give the following as the basis of the accord:

The fixing of prices in the various markets in such a way as to obtain an average return for the adherents, after taking into consideration the factors of quantity and quality, transportation charges, services, etc.

The establishment of export quotas for the three participants. The continuation of Franco-German collaboration as regards sales and the continued use of common sales bureaux, particularly in the east, with the Swiss maintaining their independent sales organisations.

The periodical readjustment of the participation of the adherents in the major market regions.

The exchange of information on methods of production and utilisation of dyes in various industries.

There is no indication of a pooling of profits.

No details have been made available regarding the plan to regulate prices, other than the statement that it is believed

that consumers throughout the world will benefit by economies to be derived by a common effort to improve packing and to rationalise distribution by effecting more direct deliveries.

#### **Sales Organisation and Markets**

The sales structure as regards the French and German members established by the original agreement in 1927 will continue. This involved the maintenance of combined sales offices in most foreign markets, with the Germans operating these sales bureaux particularly in the Far East, and with the French-operated offices in a few of the European countries—particularly Spain and Portugal. Contrary to this combination of sales effort, the Swiss will maintain independent sales bureaux in the European countries where they have already been firmly established. These countries include Italy, Spain, Czechoslovakia, and Poland, but in several of the European countries there is an elastic agreement whereby the Swiss will accept orders for the account of the French and German producers.

#### **Distribution of Markets**

In the present agreement the distribution of markets has been more clearly defined than in those signed previously between only the French and the Germans. Briefly, Germany maintains a predominance in the oriental markets, France in the South American and Latin-speaking countries, and Switzerland obtains a privileged position in the south European countries. These allotments are not final, and the Franco-German marketing collaboration may be extended even to those countries in which at the moment Switzerland holds a privileged position.

Of the three members of the entente, Germany is perhaps in the most favoured position in the Asiatic countries, because of the strong position which she has already acquired there.

Some uncertainty still prevails with regard to the Japanese markets, and the increase in production of indigo dyes in that country under the renewed subvention of the Japanese Government to the indigo industry is causing some concern to the German producers, who feel that this support weakens the effect of the German-Japanese commercial treaty. Furthermore, European producers have been feeling the effect of stronger competition from American and English producers.

It is believed now that the inclusion of the Swiss in the common sales offices in the Far East materially strengthens Germany's position because the competition of Swiss synthetic indigo in the Far East, as in several of the European countries, has cut into the sales of the German product.

#### **Exchange of Technical Information**

In view of the fact that Germany through this agreement has been able to gain a stronger position in the allotment of the export trade (75 per cent. of the combined trade of Germany, France, and Switzerland, as compared with only 80 per cent. of the trade of France and Germany), the German press has laid great emphasis upon what it considers a counter-balancing section of the accord—namely, the exchange of technical information. This is said to include not only the agreement for the reciprocal exchange of data on improved technical methods of production, but also the interchange of experience in the better utilisation of dyes, by which it is expected to improve the relationship with the consuming industries and to affect an expansion of sales. The German press maintains that the I.G. has a much wider fund of information, both as regards production and use, than have the other members, and that this concession should offset the apparent advantage which the I.G. has gained in the export field, for the reason that the French and Swiss members will now be able to employ this information to advantage in their home markets.

#### **Mont-Cenis Process to be Used in Manchuria**

AFTER trying out for three years the several current ammonia synthesis processes, the South Manchurian Railways Co. has finally selected the Mont-Cenis process, developed by Friedrich Uhde for the Hibernia-Mont-Cenis combination operating two plants in the Ruhr district. The Mont-Cenis process is in the main the outstanding ammonia synthesis in competition with the I.G.'s Haber-Bosch process. The Mont-Cenis concern, operating only experimentally a year or two ago, is now going into production with a programme of 50,000 tons of primary nitrogen this year, with prospects of increasing this to 100,000 tons. Nothing is known yet as to the size of the proposed Manchurian plant.

## **Dyestuffs Markets: The Month's Business in Review**

### **From Our Own Correspondents**

#### **Lancashire**

WITH the settlement of the cotton strike, it was hoped that the clouds over industrial Lancashire would begin to show signs of a silver lining. So far, however, a general revival of trade appears to be as far away as ever. The reduction in the cost of a piece is so small that there is still not sufficient margin between our prices and those of our competitors to influence any great volume of orders in a direction favourable to ourselves. Inquiries there have been in plenty, but they have mostly come to nothing.

The demand for artificial silk yarns, rayon as well as celanese, continues, however, to increase, particularly in yarns of 150 denier, and of 36 to 50 filaments. The finer deniers still do not meet with much popularity. Staple fibre also is not making much headway. Whilst considerable business is being done in good quality materials, the poorer qualities are only fetching a very poor price.

The Cotton Industry Research Association has announced an improvement in its income, which will now amount to close on £60,000 per annum, whilst £75,000 was stated to be the income actually required.

One of the leading figures in the dyeing trade, Mr. Adolph Hoegger, has passed away after a long illness. He founded the very successful firm of Kerr and Hoegger, which was in 1900 amalgamated with the British Cotton and Wool Dyers' Association. In 1902 he became chairman of the Association, and it is to his efforts that its great success has been due.

A settlement appears to be in sight at last of the long-drawn-out dispute over the claims of British owners of factories in the North of France which were destroyed during the war. Hitherto, the French Government had denied all responsibility, whilst the grants made by the British Government were quite

inadequate. Thanks to the effort of the Federation of British Industries, the claims of the owners are to be incorporated in the ratification of the debt agreement.

#### **Scotland**

Business in Scotland during September has only been moderate. To a certain extent the after-effects of the cotton dispute have been felt, and with the present level of business where it is any adverse influence is very quickly noticed. However, it is hoped that this effect will have disappeared by next month.

In the Glasgow and Paisley districts, dyers and printers have only been moderately well occupied. This applies also to dyers in the woollen trade.

Repeat orders for tweeds are coming through rather quicker than last month. It will be remembered that buyers were holding off because of wool prices. The manufacturers having remained firm, orders are now coming through a little more quickly.

There has been some talk of a merger among the more important firms of the Scottish woollen piece goods industry, but whether there is anything really behind this is not yet clear.

According to *The Glasgow Herald*, it is hoped next year to invite the leading wool research workers of the Dominions to a conference in this country to discuss matters of common interest, with the ultimate object of raising the standard of production and enhancing the value of the industry. The meeting will be a sequel to the gathering at the Imperial Agricultural Research Conference of 1927. Considering the economic importance of wool to the British Empire, a conference of this nature should have valuable possibilities.





## Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

*Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.*

### Colour Chemists and the Colour Council

At the inaugural meeting of the British Colour Council, reported in our last *Monthly Dyestuffs Supplement*, it was suggested—and the suggestion was warmly welcomed—that representation of the chemical and technical sides of the colour industry should be provided for on the committee. On inquiry at the headquarters of the new council, we learn that no member representing the chemical industry as such has yet been appointed. This, no doubt, will be remedied in due course, for, as Dr. H. H. Morgan and Mr. Whittaker showed, there are research and technical aspects of colour and dyeing of which the ordinary commercial man is entirely ignorant, and the committee of the new council cannot be considered fully representative until these interests are present. There is at the moment no representative of the Society of Dyers and Colourists, the Worshipful Company of Dyers, the Oil and Colour Chemists' Association, the Federation of Paint and Varnish Manufacturers, or the Paint and Varnish Research Association:

### New B.D.C. Colours

Four new and attractive B.D.C. colours are announced by Imperial Chemical Industries, namely, Acronol Green BS, Caledon Red 2G Paste, Solway Blue BGA, and Leather Green 2295.

Acronol Green BS is a new basic dyestuff, somewhat similar in general properties to the well-known Malachite Green, but possessing the important advantage of being considerably more resistant to alkalis, alkaline storage, and washing. This colour is of particular interest for the dyeing of cellulose acetate silk, as hitherto the production of bright emerald green shades has been restricted owing to the objectionable property of "fading" in the presence of alkali which characterised colours of the Malachite Green type. Acronol Green BS possesses very good fastness to light when dyed on cellulose acetate silk.

Caledon Red 2G Paste, an addition to the B.D.C. range of vat dyestuffs, is a warm-dyeing colour possessing good affinity and excellent levelling power, and is particularly valuable on account of its shade, the tone of which is at present very popular. It is specially suitable for the dyeing of cotton piece goods, although it is applicable to all types of cotton material where fastness to light and washing are required. The affinity is such that it may be used for the production of full reds, whilst good solubility and levelling power make it eminently suitable for machine dyeing.

Solway Blue BGA, a new addition to the B.D.C. range of acid alizarine dyestuffs, is of special interest for the dyeing of yarns and piece goods, as it possesses excellent level dyeing properties and fulfils all the demands for fastness that are required for these materials. It is of very good fastness to perspiration. Solway Blue BGA is particularly suitable as a component colour in mode shades.

Leather Green 2295, a recent addition to the B.D.C. range of leather colours, is of particular interest for clothing leathers. It is applicable to both vegetable and semi-chrome leathers, and the shade produced is one that is in good demand at the present time. These facts, as well as its moderate price, are expected to result in a wide use of Leather Green 2295 by firms interested in dressing sheep for motor clothing purposes.

### New Method of Dyeing Wool

Particulars have been published this week of a new method of dyeing wool direct from a simple chemical bath, without the addition of any colouring matter. The first communication on the subject was made by Mr. H. Hardy, head of the Textile Department of the Dewsbury Technical College, and unlikely, therefore, to accept the responsibility of making the announcement without having some reasonable grounds for doing so. The author is understood to be Mr. Ogden, the discoverer of the "Ogdenising" method of carbonising pure silk and cotton from wool mixtures. The chief usefulness of the discovery appears to be in the treatment of rags already dyed, but the investigations, when completed, may throw considerable light on the whole subject of the chemistry of wool dyeing—that is, the important part played in dyeing by the chemical constituent of the wool fibre. A fuller disclosure of the nature and details of the process will be awaited with interest.

### The Indigosols in Textile Printing

From Bard and Wishart, Manchester, we have received a large and beautifully produced volume on "The Indigosols applied to textile printing," issued by the well-known Basle firm of Durand and Huguenin S.A. This company is marketing, under the name of "Indigosols," a series of stable preparations, readily soluble in water, of the lenco compounds of vat dyes, which, in view of their simple and easy fixation, are specially recommended for printing and padding. Indigosol O, the first of the series, was placed on the market several years ago, and has since been followed by others that constitute nearly a complete line of colours. This volume gives particulars of the chief uses of the Indigosol dyestuffs in textile printing, with the object of popularising their use.

The subject is very well covered in the text. Beginning with a tabular survey of the Indigosols, the volume deals first with the print-on styles produced with these dyes—namely, machine printing by the steaming, nitrite, bi-chromate, and ferric chloride methods, block printing, spray printing, and yarn printing. Then follow three chapters on "Padding with the Indigosols," "Resisting the Indigosols," and "Indigosol Resists under Aniline Black." The section on artificial silk printing deals with the dyeing of viscose and acetate silks, with special reference to mixed fabrics. Silk printing and wool printing also have separate chapters. The technical data and directions are full and clear.

A feature of the book is the selection of printed samples, in very attractive designs and colouring. These show the Indigosols in machine printing, printed alongside and in combination with rapid fast colours in block and spray printing (some very striking effects) and yarn printing. There are also shown examples of white resists, colour resists with vat dyes, under Indigosol overprints, white overprint resists on Indigosol pads, coloured overprint resists with vat colours, white overprint resists alongside coloured overprint resists with ice colours, conversion styles (by Henri Schmid) produced with the aid of Indigosol O, coloured Indigosol resists under Aniline Black,

pads and overprints on mixed fabrics of cotton viscose silk, Indigosol on acetate silk and mixed fabrics containing acetate silk, on natural silk, and on wool, and finally coloured chlorate discharges on indigo. The volume is an attractive addition to any colour collection.

### A New Dyestuffs Manager

Mr. Edmund G. Robinson, assistant general manager of the dyestuffs department of E. I. du Pont Nemours and Co., Wilmington, U.S.A., since 1925, has been appointed general manager of the department, succeeding Mr. W. F. Harrington, who was recently elected a member of the executive committee of the corporation.



MR. E. G. ROBINSON

Mr. Robinson became associated with the du Pont company in 1905, upon his graduation from Swarthmore College. He was director of the Delta laboratory, chemical superintendent of the Old Hickory smokeless powder plant at Nashville, assistant director of the service department, and finally assistant general manager of the dyestuffs department. Mr. Cesare Protto, who succeeds him in this latter position, has been with the company since 1917, first in the development department and later in the dyestuffs department, where he was successively manager of technical division, assistant director and director of sales. Members of the chemical party to Canada and the United States may recall the pleasant and interesting day they spent at the great du Pont dyestuff works, under the charge of Dr. Reese.

### American Spring Colours

Advance cards portraying the eighty colours which are to appear on the regular season card for Spring 1930 have just been issued to members of the American Textile Colour Card Association. The new colours are for the most part of animated tones, though reflecting a subtlety of cast which distinguishes them from the more brilliant intense hues. Greens are accorded much importance, those with a bluish overtone like Bermuda Green being considered especially smart. The yellowish greens such as Sprig will have their place in sports wear. Several new ranges of blue are noted on the card, as this colour group is expected to provide the dominant note for spring. Tones of violet or purplish cast are outstanding.

Yellows are accented in two important ranges, both possessing considerable depth. The Japanese influence is evident in a group of mellowed golden tones called Formosa Yellow, Nippon Yellow and Imperial Yellow. The last shade is so called because it is the exact tone of the Imperial robes worn by the present Emperor of Japan at his coronation ceremony. Butter Yellow and Copa de Oro are in the range of brighter hues. Beiges continue in the fashion, but they reflect a decidedly new feeling. Beige Clair and Plage Fleurie have a subtle bisque undertone, while Custard and Baba au Rhum are golden in cast.

Much stress is placed upon brown for spring, as this range will be combined with lighter tones, such as pink, aquamarine blue, yellow, or beige, as one of the smartest interpretations of the bi-colour scheme. A new range of browns includes Milk Chocolate and Iris Brown, tones with a hint of purple in their depth. As the vogue for dahlia shades is expected to carry over for spring, two versions of purple are presented, one of more restrained amethyst tone, and the other of brighter violet hue with a suggestion of

rose. New tones in the orchid range are Wistaria Bloom and Hyacinth. Rosemist, Cloud Pink and Haze Pink are just what their names indicate; they will be especially smart when combined with the new mauvish browns. While grey does not rank in importance with other colour families, a high fashion note is reflected in a range with a mauvish rose after-tone, including Gorge de Pigeon and Iris Grey.

Two ranges of red are shown on the card, one slightly bluish in tone, and the other yellowish, thus emphasising the Spanish spirit in such names as Barcelona Red and Toro Red. Suggestive of the picturesque Basque country is a group of rust tones with a soft rose cast, including Cambo and Giroflée. A smart group of animated shades for sports and evening wear includes various hues like Violine Pink, Salad Green, Lemon Rind and Lido Blue.

### Rare Textile Museum

For several years past, Dr. Mullin, Professor of Chemistry, Rayon, and Dyeing at Clemson College Textile School, U.S.A., has been collecting rare and unusual textile materials of all kinds from various parts of the world. During the past two years he has collected many very interesting samples of weaving, dyeing, printing, finishing, etc., mostly from Europe and Asia. While particular attention has been given to the chemistry and dyeing branch of the textile industry, other phases of the subject have not been neglected, and one of the latest additions includes about eighty pictures woven from the finest silk. Each of these pictures is just as perfect as if it were painted with a brush or printed, but it is actually woven in the cloth on some form of jacquard loom, probably by hand. European artists who have examined this collection of silk pictures state that, from an artistic standpoint, the pictures are wonderful, and would be excellent work even if they were painted with a brush instead of woven. While most of the pictures are in black and white, a few are woven in colours.

On account of the often insurmountable difficulties encountered in transferring a painted picture into a loom design, the fact that these pictures are woven makes them all the more wonderful and especially interesting to all connected with the textile industry. In fact, it takes a weaver or designer to appreciate the difficulties of producing work of this kind even on the hand loom. As this collection will form the nucleus for a textile museum at Clemson College, Dr. Mullin would be glad to hear of any textile or related samples suitable for the collection.

### United States Dyestuff Imports

The following table shows the imports into the United States of coal-tar dyes for 1928 (preliminary figures) and for the years 1920 to 1927, together with the imports for the fiscal year 1914. The quantity for the year 1914 was taken from the Department of Commerce, Special Agent Series 121, and the value from Commerce and Navigation. The figures for the year 1920 to 1927, inclusive, were taken from the Annual Census of Dyes and Other Synthetic Organic Chemicals, published by the United States Tariff Commission. The final figures for the dye imports of 1928 will be published in the Census of Dyes and Other Synthetic Organic Chemicals for that year:—

Year.	lb.	Value.
1928 .....	5,348,227	\$4,322,621
1927 .....	4,233,046	3,413,886
1926 .....	4,673,196	4,103,301
1925 .....	5,209,601	4,637,240
1924 .....	3,022,539	2,908,778
1923 .....	3,098,193	3,151,363
1922 .....	3,982,631	5,243,257
1921 .....	4,252,911	5,156,779
1920 .....	3,402,582	5,763,437
1914 .....	45,950,895	9,502,714

## The Story of Scottish Dyes, Ltd.

### What British Chemists Have Achieved

THE story of Scottish Dyes, Ltd., the difficult circumstances in which the company was established, and the achievements that stand to its credit in the field of dyestuffs production, is told in simple terms in the November number of the *I.C.I. Magazine*.

Scottish Dyes, Ltd., it is stated, is one of the youngest members of the I.C.I. family. It does not go back any further than 1914, and in fact, under its present name, only as far as 1919, and so, unlike some of the I.C.I. companies, it lacks the changing circumstances of a hundred years or more on which to draw for historical material. However, the story of its birth is an exceptionally interesting and even romantic one, and the fifteen years of its youth, which have

that the special colours were fast and the ordinary ones fugitive.

To appreciate the reason for the use of these colours it is necessary to go back for a moment into another history, that of the Morton textile concerns. In the eighteen sixties, Alexander Morton, a young Scotsman, started a weaving industry in the Irvine valley, and the industry thrived because of the widespread reputation for quality gained by his goods. He died two or three years ago, and his name is now remembered by its inhabitants as the man who led the Irvine valley to industrial fame and prosperity. In the year 1900 he had also started a works at Carlisle. Here, among other things, tapestries were woven, and Mr. James Morton, his son, was responsible for the colour schemes. One day in 1902, looking into Messrs. Liberty's, of Regent Street, he saw a display of these tapestries, but all his colour schemes were spoiled by the fading of the colours. Now fading was so well-known that it was more or less accepted as inevitable. This, however, did not reconcile him to the spoiling of the curtains, and instead of bowing to the inevitable, he immediately began to collect cuttings of all the coloured materials, both English and foreign, on which he could lay hands; these he exposed in hundreds in a greenhouse, first covering up half of each cutting to serve as a reminder of the original shade. The results of these tests were appalling—only here and there did a colour show any real resistance to sunlight. He picked out these few, and, unpromising as they were for a start, he determined to make them the nucleus of a new series of tapestries which would not fade.

To-day, about twenty-five years later, it is almost a commonplace that curtains and other materials can be bought, which can be put up in the window and exposed to blazing sun without fear of spoiling. For this great advance Mr. James Morton, more than any other one man, is responsible.

#### The Campaign Begins

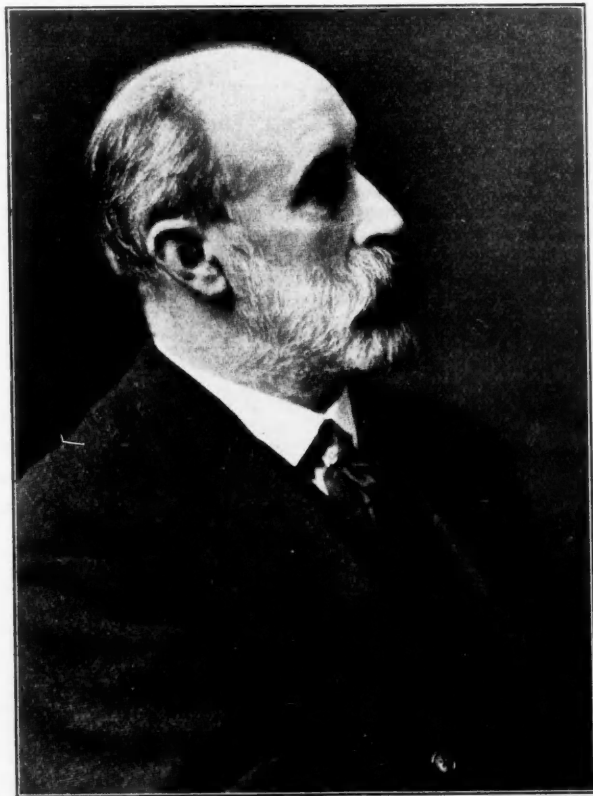
In this early campaign for getting fast materials two points may be mentioned, first that Mr. Morton arranged for the firm to do its own dyeing, and, secondly, that the German dyestuff makers started a new series of colours now known as the Indanthrenes. These Indanthrenes had a fastness quite out of proportion to any other series known before. They came as a very great help in making these guaranteed fabrics. At the same time the manufacture of these fadeless materials gave a considerable impetus to the manufacture of the new series of fast colours which, strange as it may seem now, were not adopted for several years after their discovery.

Now, turning back to 1914, it will be clear why the stopping of these fast dyes came as such a calamity to Morton Sundour Fabrics. Without the proper dyes all the work of the previous ten years was in jeopardy.

#### The Birth of Scottish Dyes

However, instead of being daunted, Mr. Morton decided that as he could not get the dyes he would try to make them himself, although he knew nothing about the work. He thought if he could only manufacture a blue and a yellow, that by reverting to some of the dyes which he had used in his first fast cloths, he would be able to continue with a limited range of Sundour goods. This determination to make dyes himself marks the birth of Scottish Dyes. With the aid of the head dyer he managed to learn sufficient about the new blue and yellow to find they were made from anthra-quinone beta-sulphonic acid, a body which was entirely unknown to either of them.

Some of this substance was bought from another firm, and, after very hard work, by early November, 1914, a little of both Indanthrene Blue and Indanthrene Yellow were made. The next stage, producing in larger quantities, required further courage and resource, as the process had to be done under high temperature and pressure, and apparatus for this had never been made in the country. However, it was finally done, and soon they were making the two colours in quantity and using them. Then one or two other colours were added to the programme, and later there was enough being made to spare some for other manufacturers.



MR. JAMES MORTON, FOUNDER OF SCOTTISH DYES, LTD.

coincided with fifteen very important years in the development of the British chemical industry, have been filled with struggle and achievement.

The story opens when supplies of German dyestuffs had ceased on the outbreak of the war. Nine out of every ten pounds of dye used in Great Britain had been coming from Germany, and the effect on the textile industry—a very important one, making £200,000,000 worth of goods annually—was extremely serious. Among the manufacturers affected was Mr. James Morton, the founder of Morton Sundour Fabrics, Ltd.

Almost immediately, the dyemakers who had already been established in this country before the war set to work to increase their output as much as possible. The Sundour fabrics, however, required special dyes, the latest and best products of the German chemists. These dyes were not well known; they were not the type for which dyers were clamouring and which the manufacturers would be making first. Unfortunately for the Sundour fabrics, it was not possible to change from the special colours to the ordinary ones, the reason being

So far, the dyemaking had been, in a sense, a branch of Morton Sundour Fabrics, but with the sales to outside firms continually increasing, it was on July 1, 1917, detached and made into a separate company, Solway Dyes, Ltd.

With the formation of the new company the range of fast dyes of the "vat" series was extended. These colours were given the name "Caledon" colours. The Caledon colours are now extremely well known, and renowned for their fastness.

It was in January, 1918, that Dr. John Thomas joined the firm from Nobel's. He was soon made first director, and then managing director, and in the very difficult years which were to follow proved fully equal to his task.



DR. JOHN THOMAS.



MR. G. W. SHEARER.

Mr. G. W. Shearer also came to the firm in 1918, and has been in charge of the engineering ever since, and in addition to the plant has also been responsible for the new extensions.

In 1918, the prospects were brighter than they had ever been before, and new chemical houses, offices and laboratories were erected in Carlisle. Even these, however, were not sufficient to meet the rate at which the business was expanding, so an even larger works was started at Grangemouth and a new company—Scottish Dyes, Ltd.—formed. Soon after this new prospect was launched the outlook changed altogether—following in quick succession came the end of the short war boom, the Sankey judgment, and the coal strike; then, with one factory already sufficient to meet the highest previous sales figures, and another half built, the demand for colour dropped to practically nothing. As this state of affairs persisted, the very existence of the company was threatened. However, although production was almost at a standstill, the

management took a long view of the situation, and the chemical staff was temporarily diverted to research work.

As an outcome of this research a new colour of the Caledon series, now known all over the world as Caledon Jade Green, was discovered. This was a very important advance, as among the fast colours there had never been a proper green, in spite of many determined efforts on the parts of German manufacturers to discover one. This new green was not only remarkably bright, but its fastness properties were also irreproachable, and the combination of brightness and fastness created a good impression amongst colour chemists, brought Scottish Dyes prominently before the chemical world, and also showed that British manufacturers could make colours of their own as well as successfully reproducing the old colours.

Meanwhile, as a result of the Sankey judgment, enormous quantities of German dyestuffs were still coming into the country. In order to prevent Scottish Dyes and the other British manufacturers from being ruined, strenuous attempts were made to get an Act through Parliament which would help to preserve the new dye industry which had been built with such difficulty during the War. As a result, the Dyestuffs (Import Regulation) Act was passed. The stocks of colour which had accumulated were too large for immediate effect to be felt, and trade conditions remained poor, so that although improvement began, it was only felt very slowly, and the position remained one of great difficulty for several years.

#### *Developments at Grangemouth*

However, to return to the growth of the Grangemouth works—at the beginning of this period it had been seen that there was not enough room at Carlisle for continued expansion, and therefore a site for additional works was chosen at Grangemouth, with the intention of making this the main works.

Grangemouth is situated midway between Glasgow and Edinburgh, in the centre of the industrial area of Scotland, and is convenient for transport by rail, sea and road, or even canal. But, in spite of this, the works are, to all intents and purposes, in the country, and across the road from the main entrance is Kerse House and its grounds, once the residence of the Marquess of Zetland.

The works were designed by the late Sir Robert Lorimer, the Scottish architect, who designed the Scottish National War Memorial in Edinburgh Castle. The factory is well laid out, and building began in 1919.

When complete, the works were declared the finest of their



INTERIOR OF ONE OF THE CHEMICAL HOUSES.

kind in the country. Erection was delayed to some extent as an indirect result of the moulders' strike, and in the first three years, as has already been stated, the company was having a particularly hard fight in every way. However, as each building was finished, it was nevertheless found possible to start operations, and, moreover, it was soon found necessary to make additions to the original scheme.

Extensions have proceeded ever since, and the whole is now roughly twice as big as originally intended. So great has been the call for extra space for new processes that, while it was originally desired to have the operations at Carlisle removed to Grangemouth by the end of 1920, now, nearly ten years later, the transference is not yet quite complete.

Up to 1926 Scottish Dyes had worked as an independent firm, and while some of the same fast colours had also been made by British Dyestuffs Corporation these were not one of

their principal lines. By this time, however, the importance of these fast colours was becoming so great, and promising to become so much greater still, that a firm of the importance of British Dyestuffs could not safely leave them in the hands of a competitive firm. As a result of negotiations an arrangement was made to avoid this unnecessary duplication. British Dyestuffs acquired a majority of the share capital, and the manufacture of these fast colours was concentrated at Grangemouth under the original management. At the same time, Mr. James Morton joined the board of the Corporation and Professor Sir William Pope, who had previously been retained in a scientific capacity, was offered and accepted a seat on the board of Scottish Dyes. Subsequent developments have shown the wisdom of this arrangement. Later, with the formation of Imperial Chemical Industries, Scottish Dyes became a constituent company.

## **The Laundering of Woollen Goods**

**A Lecture by Mr. R. G. Parker**

*At the Clothworkers' Hall, London, a lecture was recently delivered on "Woollens from the Laundry's Point of View," the lecturer being Mr. R. G. Parker, B.Sc., F.I.C., Director of Research to the British Launderers' Research Association.*

*The lecture was given under the auspices of the London Section of the Textile Institute.*

MR. PARKER said that wool was an important product of the British Empire, and, quite apart from the home-grown product, constituted in 1928 one-fifth of the imports of raw material into the United Kingdom. Although much of that raw material was manufactured into suitings and other non-washable goods, a great quantity of washable articles of all descriptions was produced. The washing and re-finishing of textile fabrics was by no means a negligible matter from the point of view of those engaged directly in the textile trades, or connected with them. In the past the matter could hardly be said to have had serious consideration; but, fortunately, that was no longer so.

### **Co-operation**

The spirit of co-operation between industries, so marked a feature of post-war development, was at work, and through their respective research associations, the textile trades and the launderers were approaching one another, and had already aided one another in their investigations.

The importance of the matter to the nation was very great; it was, in fact, easily under-estimated. Textile fabrics were, compared with other manufactured articles, of a fragile nature; in ordinary use they showed progressive deterioration throughout their life, however carefully handled. Chemically, they were composed of complex organic substances, by no means inert; and being built up of fine fibres, they presented an extremely large area towards any reagent, gaseous or liquid, in which they might be placed. Neglecting exceptional conditions, or even ordinary accidents likely to occur frequently in use, the fabric was exposed to the action of light, air and moisture; and in washing it was in contact with aqueous solutions at temperatures up to 212° F. for cottons and 120° F. for wool.

No change which might occur in the fabric increased its strength or assisted in prolonging its life. The methods of treatment which attained the end in view while they ensured a minimum rate of change were therefore to be sought. It had been estimated that an extension of the life of the laundered fabrics by so little as 5 per cent. in respect of about one-third of the laundries of the country would result in an annual saving to the public of some £100,000.

### **Handling in Bulk**

The lecturer went on to say that it was essential that the objects in view should be attained by processes suitable for handling of goods in bulk. In the laundry industry, the present cost of labour rendered any other methods impossible of employment, and over 50 per cent. of the cost of running a laundry was represented by wages and salaries. The great bulk of the articles received for laundering consisted of white undyed cotton goods.

As regarded retention of strength, cotton and linen were more than satisfactory, because they increased in strength when wet. Compared with cotton or linen, wool was a much more complex material, and chemically its composition was

such that it was much more active. It was much more sensitive to alkalis, to heat, to bleaching agents and to impurities in water, and it was much more retentive of impurities when once it had absorbed them. So much so, that it was not difficult to find old woollen fabrics which no practical treatment would improve. Fortunately, wool did not retain solid particles of dirt any more vigorously than cotton, but appeared to part with them rather more easily.

### **Properties to be Kept in View**

From the launderer's point of view, the main properties of wool which they must keep constantly before them were liability to shrink and felt, power of absorbing impurities (leading to loss of colour and handle), and weakness when wet. With cotton, they were loss of strength, loss of fibre, and loss of colour. The differences between wool and cotton made it necessary for the washing process to be so altered as to cut down mechanical motion as much as possible.

The main precautions to be observed in selecting materials for laundering woollen fabrics were that the soap should be freely soluble and neutral, and not contain resin or other impurities. The water should be free from hardness, and, still more important, free from heavy metals such as iron and copper. Some alkali should be used with the soap, in order to hinder the hydrolysis of the soap by the wool, and thus lessen the absorption of fatty acid. The alkalinity must be much less than for cotton goods, or it would promote felting and yellowing. It was more convenient and safer to provide that alkalinity by mild substances such as borax. The water used was usually of zero hardness, having been softened by the zeolite process, or in some cases distilled.

The absence of calcium compounds was important, as they readily loaded the fabric, producing greyness and yellowness. But the absence of iron was much more important, and great care must be taken that the water had not been allowed to pick up iron (e.g., by passage through rusty pipes and tanks) after having been softened. Deterioration of the colour of bleached white wool might take place as follows: (1) The wool might develop a yellow tint after successive washes, and stains accidentally produced might not have been removed in the wash; (2) greyish and yellow-grey tints might appear, and upon examination the wool was found to contain ordinary soap, fatty acid, acid soaps (which might be regarded as mixtures of ordinary soap and fatty acid), lime soaps or aluminium soaps; and (3) the wool might absorb iron or copper.

### **Colour Considerations**

The colour of bleached white wool, especially if the sulphur dioxide process had been used in manufacture (wool bleached by hydrogen peroxide was to be preferred), slowly deteriorated, even in the absence of definite impurities, becoming yellow and dull. It had to be restored by careful treatment, bleaching as in manufacture, but more gently. Hydrogen peroxide was the best all-round agent for laundry use, and could be

employed without producing any alteration in the handle or strength of the fabric. It was used in the cold, and must in any case be employed if stains were present which had proved resistant to washing. Apart from accidental stains, such bleaching need only be given once per six to ten visits to the laundry to maintain excellent colour.

#### **The Launderer's Task**

The launderer had no easy task to perform if he was to handle successfully large quantities of woollens which must be ready for return to their owners in a few days at the outside. They were less robust than the cotton goods, and, though stronger than many artificial silk fabrics when wet, liability to felting placed them at a disadvantage when compared with those materials. By co-operation with those who had to cleanse their washable woollen fabrics, manufacturers and suppliers might render a great service to themselves, to launderers, and to the public as a whole.

The guiding principle throughout was that the fabric, whether of the first quality or of a cheaper grade, must be honestly the most suitable for its particular purpose that could be supplied. If the price offered was low, so as not to permit of the supply of the quality and kind of article definitely required for the work, then it would be better for all parties if it were not supplied at all. The position was a difficult one, since there was always a demand for cheap goods, and these might be unsuitable for use. For example, treatment to render woollens unshrinkable added to their cost; there must, therefore, be a tendency to supply crossbred hosiery fabric of natural finish for cheaper grades of underwear. The consumers of that type of article would be largely of the working classes, and manual labour, etc., would cause the articles to be heavily soiled.

Here there were three factors militating against the continued useful life of the fabric—it was heavily soiled, it was less ready to part with its dirt than fabrics of finer yarns, and it was unable to withstand, without felting, the vigorous treatment by which alone it could be cleansed. If it had been possible to add the cost of unshrinkable treatment, the position would have been quite different.

#### **Important Points**

Some important points were that washable fabric should always be of unshrinkable finish, unless for some good reason; it should be sold to the public in such a condition that no shrinkage took place upon mere immersion in warm water or warm detergent liquor; dyed fabrics should at the very least be fast to the washing process described, and the strength of the warp and weft should not differ so greatly from one another that failure readily occurred in one direction only. In various details the manufacturer and supplier could assist the launderer, who in turn could then so launder woollen goods as to satisfy the user.

#### **Wool and Worsted Research**

A MEETING of members of the British Wool Federation at Bradford decided almost unanimously to support a proposal which has been under consideration for some time by this and 11 other federations in the wool textile industry for making two voluntary levies in support of the British Research Association for the wool and worsted industries, the headquarters of which is at Leeds.

The first levy would be of twopence on every bale of wool entering the country and retained for home consumption. The second levy would be one of a farthing per week per operative employed. The first levy would apply to the raw material end of the trade, and the other to the manufacturing and semi-manufacturing sections. The levies are estimated to yield £10,000 each, and the Government has agreed that if the trade can raise £10,000 it will make an equivalent grant to the association of £1 for every £1 raised over that figure—the maximum grant to be £10,000.

The Government considers the minimum effective expenditure of the association should be £30,000, and the proposed levies and the Government grant would provide that amount. The executive committees of seven of the federations have approved the scheme, and five others have yet to report. The British Wool Federation is the first federation to take a vote of its members on the subject, and the decision supports the recommendation of the executive.

### **Direct Dyeing of Wool**

#### **A New Chemical Bath**

CLAIMS on behalf of a discovery for dyeing wool direct from a simple chemical bath without the addition of colouring matter were advanced at Dewsbury recently by Mr. Harry Hardy, head of the Textile Department of the Dewsbury Technical College, in a lecture on "Scouring and Milling" to members of the Dewsbury and District Cloth Millers and Scourers' Association. Mr. C. F. Archer (Morley), president of the Association, presided.

Mr. Hardy said his announcement would specially interest those concerned with the chemistry of dyeing. A method of dyeing wool direct from a simple chemical bath, without the addition of any colouring matter, had recently been discovered, and, for the past few weeks, he had been co-operating with the discoverer. Remarkable results had been obtained.

The patentee, whose permission to make the announcement had been received, was already fairly well known to chemists in the textile trade, through his discovery of a hydro-cellulose with peculiar properties. He was, however, better known as the discoverer of the "Ogdenizing" method of carbonising pure silk and cotton from wool mixtures, to which his name, Ogden, was lent.

#### **A Simple Method**

By means of this newly discovered chemical bath, simple immersion for a few minutes was sufficient to convert white wool or natural silk to a light yellow of exceptional brightness, longer immersion deepened the colour until it became a deep orange, and any intermediate shade could be obtained by varying the time or concentration of the solution.

The chief usefulness of the discovery appeared to be in the treatment of rags which had already been dyed. Dark or faded blues and blacks, or even dark fancy worsteds or dark fancy merinos—irrespective of colours—were converted to yellow or red-brown, light brown or deep chocolate in a single bath within a few minutes. Indigo was also affected, so there were practically no "throw-outs," and colour blends could be effected without sorting.

A feature of the process, added Mr. Hardy, was that there was little or no bleeding of colour in the bath during conversion, evidenced by the fact that there was no loss of weight in the material in this respect, as occurred in the usual stripping and after-dyeing treatment. This fact, together with the cheapness of the chemical and the relatively short time required to dye material, should reduce very considerably the costs of dyeing rags or shoddy.

As might be expected from a process where the colouring matter combined with the chemical constituent of the wool fibre, it was fast to all ordinary influences. Severe milling tests and tests for fastness to light had proved satisfactory. In milling, the accompanying white wool and cotton were unstained. Cotton or artificial silk present during dyeing remained unaffected, but natural silk was dyed the same as the wool.

It was expected that when investigations had been completed, a great deal of light would be thrown upon the much discussed subject of the chemistry of wool dyeing, namely, the important part played in dyeing by the chemical constituent of the wool fibre.

#### **Dyestuffs Licences for October**

THE following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during October, has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—

The total number of applications received during the month was 694, of which 628 were from merchants or importers. To these should be added 18 cases outstanding on September 30, making a total for the month of 712. These were dealt with as follows:—Granted, 674 (of which 645 were dealt with within 7 days of receipt); referred to British makers of similar products, 36 (of which 28 were dealt with within 7 days of receipt); outstanding on October 31, 2. Of the total of 712 applications received, 673 or 95 per cent. were dealt with within seven days of receipt.

# Dyes and Their Application: Recent Technical Progress

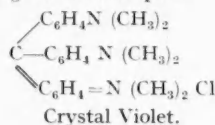
By L. J. Hooley

In the October issue, azo-mordant dyes were considered, especially those of the type where the metal is combined with the dyestuff before use. Another type of metallic compound which has not previously been dealt with is that formed from dyes, especially basic ones, with complex acids of phosphorus, tungsten and molybdenum. These are primarily pigments and lakes rather than dyes.

## Fastness Plus Brightness

The basic dyes have the advantage of brightness and high colour value, but are weak in light fastness, and although the light fastness can be improved by after-treatment with copper, this spoils the brightness; any process which will give increased fastness without spoiling the brightness is therefore of interest.

In 1913, the Badische Anilin-und Soda-Fabrik (G.P. 286467) found that colour lakes could be formed from basic colours with phosphotungstic acid. The method of preparation consisted in adding a solution of the sodium phosphotungstate to a mixture of dye solution with a substratum such as alumina and blanc fixe; Crystal Violet, which gives a pure violet colour, Rhodamine, Victoria Blue B, Diamond Green and Auxamine were given as examples.

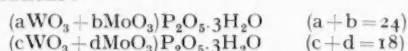


In the following year it was found that other phosphotungstic acids, as well as other complex acids—silicotungstic, phosphomolybdic—could be used as well. The formation of these lakes was found greatly to increase the fastness of the basic colours used.

## Printing Inks

During the war the development of the process was delayed, but further work has been carried out since, and can be seen from the following British patents (all from the I.G.F.A.-G.), while the products have now been put on the market for printing inks as Fanal colours.

One of the first developments was the discovery that instead of mixing in the dye with the base, the substratum, and the various components of the complex acid, the last could be first prepared by itself. This preparation is described in B.P. 216486 (Bayer), by mixing solutions of sodium tungstate, molybdate and phosphate, the resulting acids being precipitated by the addition of mineral acids. Silica may be substituted for phosphorus with the production of analogous acids. These complex acids are then added to the solution of the dyestuff and substratum. The acids have the following constitutions:—



Those of the first series are less soluble than those of the second, and separate first in the preparation.

In B.P. 265032, the lakes are prepared from other complex tungstic acids (excluding those containing molybdenum) and another acid, with or without a substratum. In an example, a substratum is made by mixing heavy spar with sodium sulphate solution and precipitating with barium chloride. A solution of the dye is then added, followed by solutions of sodium tungstate and phosphate and then hydrochloric acid, and on heating to 80° C. the insoluble lake is formed and precipitated.

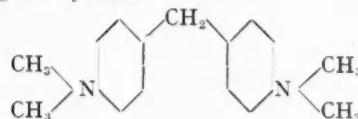
The above processes may, if desired, be carried out with basic dyes containing sulphonic acid groups.

## A Pressure Treatment

In B.P. 270750, a pressure treatment is introduced, this being at a high temperature and carried out either during or after lake formation. It increases the fastness to light and alkalis. Temperatures of about 50–100° C. and pressures of two or three atmospheres are used.

The starting materials for these complex pigments need

not necessarily be dyes. Coloured compounds such as aromatic amines, e.g., 4,4'-tetramethyldiaminodiphenylmethane, which are not dyes can also be used (B.P. 275943). From the amine just mentioned a deep blue is obtained with sodium phosphotungstomolybdate.



The corresponding benzhydrol gives a deep bluish violet and the ketone an orange brown. *p*-Amino benzaldehyde, dissolved in dilute hydrochloric acid and mixed with sodium phosphotungstomolybdate, gives an orange red.

In an addition to this process, the amines above mentioned may be used in conjunction with basic dyes, thus Victoria Pure Blue BO, Michler's hydrol and sodium phosphotungstomolybdate give a deep blue (B.P. 299,521).

In B.P. 275969, these complex lakes are used in cellulose esters or other lacquers. The dyestuffs are added along with the necessary complex acids, either in solid form or in organic solvent.

## Application as Dyes

The application of the products as dyes as distinct from pigments is seen in B.P. 283,281, where the complex acid is fixed on the fibre and then the dyestuff applied. The acids themselves need to be first fixed on the fibre, and this is done by the help of amines and phenols, etc., or by means of a reducing agent. As an example, phosphotungstomolybdic acid is applied to cotton with the aid of benzidine hydrochloride, and the cotton thus treated is then dyed a fast violet with the product from Michler's hydrol and dibenzylanilinedisulphonic acid. Either basic or acid dyes may be used in the process.

Another method of using these complex pigments as dyes is to reverse the above procedure and apply the dye first and the complex acid second. The material is dyed with basic colours in the ordinary way, and then treated with reduction products of the complex phosphotungstomolybdic acids. Thus Rhodamine B dyed on a tannin mordant in the normal manner is aftertreated with a solution of the reduction product and dried (B.P. 294,286).

## Auxanin B

In this connection Auxanin B, which was put on the market recently, may be mentioned. This is described (P. Rabe, *Textilb.*, 1928, 665) as a violet-black product soluble in water. It is used for after-treating goods dyed with basic colours, and improves the fastness to light, bringing this into Class 3 or 4. The method of treatment consists in treating the goods, already dyed with basic colours on a tannin or Katanol mordant, in a 2.5 per cent. solution of Auxanin B for ½–¾ hr. at 25–30° C.

A new series of acids have recently been obtained by treating those already mentioned in connection with B.P. 216486 with reducing agents, two new parallel series being formed. These reduction products are stable, but can be reconverted to the original ones by permanganates or other oxidising agents. Sulphurous acid, bisulphite or hydrosulphite may be employed in the reduction. These reduction products are the ones which are used in B.P. 294286.

## New Consular Appointment at Maracaibo

THE Department of Overseas Trade announces that Mr. T. J. Anderson, who has been appointed to be H.M. Consul at Maracaibo, Venezuela, will be available at the offices of the Department during the period November 11 to 30, inclusive, to interview, by appointment, manufacturers and merchants interested in the export of British goods to Maracaibo or to the Dutch colonies of Curaçao and Aruba, which will be included in Mr. Anderson's consular district. Firms desiring interviews with Mr. Anderson should apply at once to the Comptroller-General, Department of Overseas Trade, 35, Old Queen Street, London, S.W.1, quoting reference No. 1942/29.

### Cutting the Cost of a Dye

ONLY four short years ago, states *Chemical Markets* (New York), a certain imported dye was selling in our domestic markets at 35 cents a pound, duty paid, while the best cost of the largest American manufacturer was well over one dollar a pound. If we might make a million pounds—so the operating chief reported—we might then be able to bring the cost down to within the neighbourhood of half a dollar, which was obviously impractical and not extremely helpful. As both the American and foreign processes were substantially the same, and employed the same raw materials, it seemed clearly either a chemical miracle or a plain case of dumping. Within the past six weeks the same American production department has gleefully recorded a cost for the identical dye made by the same old process of 20 cents a pound.

This is no industrial fable related for the sake of its pretty moral. The whole story was told quite recently to a small group of chemical men at lunch by the American company's president, and there is, moreover, no chemical manufacturer of any considerable experience who cannot remember equal astonishing results of the "know how" in chemical production. It was no epoch-making discovery that clipped four-fifths off the cost of that dye—a change in time here, closer temperature control there, a new bit of apparatus somewhere else, all aided no doubt by a little saving in labour and a lower cost of some of the materials, all combined to work the seeming miracle.

These little tricks of the trade have great effects upon the chemical industry. Seldom do they get into even casual, friendly conversation; they never appear in our technical literature. They are scrupulously omitted from all patent specifications. They are, however, most dearly cherished secrets, since they give their fortunate possessor a fundamental advantage over each of his competitors. Thus, they foster that secretive spirit which appears to outsiders so unpleasant and unprogressive a characteristic of the chemical industry. At the same time they inject variations in production costs which colour chemical selling problems blue and chemical balance sheets red. These kinks of "know how" are an element inherent in chemical manufacturing; and if they cannot be eliminated, we must be prepared always to reckon with their results.

### Delustring of Cellulose Acetate Rayon

DISCUSSING the delustring of cellulose acetate crêpe fabrics, a special correspondent of the *Rayon Record*, writing in a recent issue, states that—"When cellulose acetate rayon materials are free from tension they commence to delustrate readily in soap solutions maintained at a temperature of not less than 90° C. As the tension is increased, so does the

minimum temperature increase at which delustring commences. Further, the rate at which delustring takes place increases rapidly with increase of temperature. Thus when the crêpe fabric must also be delustred the temperature of the soap solution is suitably increased. In a limited number of instances almost complete delustring is desired, but in the majority of cases only partial delustring is necessary. It is obviously difficult to obtain regularly a specified lustre when the delustring treatment is also closely bound up with the crêping effect, but it is possible by careful control of the conditions of temperature and duration of treatment. Practically no control is possible by varying the concentration of the soap liquor, since it requires but a very small proportion of soap to enable full delustring to be obtained. Use, however, can be made of the discovery of A. J. Hall (E.P. 246,879) that the delustring of cellulose acetate rayon in boiling aqueous liquors is retarded by the presence of inorganic salts. It is found that delustring in the above processes can be regulated within definite limits by adding appropriate amounts of such substances as sodium chloride, and particularly potassium chloride, to the soap solutions employed. If too much of these substances is used no delustring takes place at all."

### Dyers' Wages

MR. T. D. BUTTERCASE, a director of the Bradford Dyers' Association, Ltd., and head of the Association's Labour Department, stated in an interview on Thursday, October 31, that he understood that the Amalgamated Society of Dyers were considering certain proposals which the Association had put forward with a view of reducing their wages costs. "There is nothing new in the efforts of the B.D.A. to reduce costs of production," he said. "This is going on all the time, and it is obvious in the circumstances at present prevailing that it is more imperative than ever to concentrate on the reduction of costs. In these efforts we naturally seek the co-operation of the trade union leaders and our employees." He added that he was not disposed to quarrel with the suggestion that the proposals were concerned with efforts to increase production by arranging for operatives to tend more machinery than they did at present.

A meeting of the Amalgamated Society of Dyers is believed to have been held on Saturday, November 2, to consider the matter. Operative dyers—including those employed by the Bradford Dyers' Association, Ltd.—are to a great extent outside the jurisdiction of the Joint Industrial Council for the wool textile industry, and are not, therefore, involved in the prevailing wages dispute, but they are apparently to be affected by separate proposals to reduce costs of production in the works of the Bradford Dyers' Association.

## Dyestuffs Markets: The Month's Business in Review

### From Our Own Correspondents

#### Lancashire

THE cotton trade in Lancashire has hardly yet recovered from the effects of the strike; that is to say, dyers during the past month have still been hampered for want of both yarns and pieces. Trade is very quiet in piece-dyeing, where one of the largest combines is finding it difficult to keep its Lancashire works running anything like full-time. The calico-printing section is also very quiet.

As far as dyestuff prices are concerned, it can hardly be said that these are fully stabilised. By yarn dyers especially, pressure is being exerted to bring down prices of dyestuffs, on the plea of potential business. October appears to have been a very quiet month with all the colour-makers.

One or two new products are being introduced; notably, Acronol Green BS, for which the important property is claimed that it is faster to alkalis than Malachite Green, which, in this regard, is most unsatisfactory. This dyestuff is announced by I.C.I., who also report the addition to their range of vat dyes of Caledon Red 2G, of good all-round properties. From the Clayton Aniline Co., comes news of a new Viscose Navy Blue, which, especially for dyeing viscose and cotton unions in solid shades, is expected to meet a real want. The Icy range has been extended by the introduction of Icy

Blue 2RS. This dyestuff is recommended for application to viscose artificial silk, and it is claimed that it gives even dyeings on material of irregular quality. It possesses a reddish-blue shade, is of good fastness to washing, and is dischargeable to a good white by reducing agents such as Rongalite.

#### Scotland

Business in Scotland has been rather patchy during the month. As far as cotton is concerned, the effects of the recent stoppage do not appear quite to have disappeared, and woollens are affected by the falling wool market. The Calico Printers Association printworks at Bridge-of-Weir are now stated to have closed down altogether, work being concentrated at branches such as Thornliebank.

Round the Glasgow district dyers and printers have not been so badly off for orders, being in fact somewhat busier than during recent weeks, but dyers and printers elsewhere have no improvements to report.

The tweed manufacturers of the Border are still fairly busy, being occupied principally with spring orders for 1930.

Sales of dyestuffs are fairly steady, with slight improvements in the vat and direct sections.





## Dyestuffs Monthly Supplement

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### The Great Patent Action

THE great patent action between Imperial Chemical Industries and the I.G. Farbenindustrie is still proceeding in the High Court of Justice before Mr. Justice Maugham, with Professor Smithells as chemical assessor. Up to the time of going to press the hearing has already occupied 15 days. Mr. Whitehead's opening address covered four days, Dr. Oberlander's evidence and cross-examination 9 days, and Dr. Goldsmidt was in the witness box for 3 days. The I.C.I. case has only just been opened. At the present rate of progress the action promises to run well into the New Year, and in an issue of so much importance there is always the possibility of appeal proceedings. The case, judging from the attendance in court, does not appear to be attracting much public interest, the hearers consisting of the witnesses and a few chemists.

### The Dyestuffs Report

The latest information relating to the report on the working of the Dyestuffs (Import Regulation) Act is that considerable progress has been made. The Act has still twelve months to run before the period of "ten years and no longer" for which it was passed expires, and naturally before that point is reached any Government that might be in power would desire to have an authoritative review of its working and results together with the views of the interests immediately affected. It is not in doubt that any report must bear witness to the very remarkable progress in the whole science and practice of dyestuffs manufacture that has taken place since the Act was passed, and to the fact that progress has been made possible by the protection—using that word in its ordinary meaning—against unlimited world competition that the Act has afforded the industry in its infant stages. The questions that remain are whether that protection at the end of 1930 should cease altogether, or should be continued on the present lines, or should be modified in proportion to the internal strength of the industry. These are points on which the report may be expected to throw light and to supply facts and reasons for forming a sound judgment.

### Fashionable Shades for Spring

Imperial Chemical Industries, Ltd., have just issued, through the British Dyestuffs Corporation, what is described as the Fashionable Shade Forecast for Spring, 1930. The selected colours number ten, and they are offered merely as suggestions, the B.D.C. being prepared to give recipes for the matching of any other desired shade on wool or any other material. The dyeing of the colours is carried out in the usual manner for acid colours with the addition of 10 per cent. Glauber's salt and 2 per cent. sulphuric acid. The shades include several of a stronger and more distinct character than those in vogue last year, but the delicacy which has recently characterised popular fashion tints has not yet disappeared. The ten colours are:—Surf, a palish green produced from Lissamine Fast Yellow 2GS, Lissamine Fast Red BG and Solway Blue BGA; Sand, a light fawn, produced by the same range of colours in different proportions; Lilac Time, a tone which well agrees with the selected name, produced from Solway Blue and Fast Acid Violet 2RS; Oriole, a very brilliant and striking orange, produced from Lissamine Fast Yellow 2GS and

Acid Orange 2GS; Raisin, a deep rich tone composed of the same colours as the first two named; Myosotis, a clean blue tone from Solway Blue RS; Wild Rose, a tint agreeing with its name, produced from Lissamine Fast Red BG; Nightingale, a good brown produced from the three colours in combination already mentioned; Malachite, a good reproduction of this well-known colour from Lissamine Fast Yellow 2GS and Solway Blue BGA; Red Pepper, a brilliant red, obtained from Lissamine Fast Yellow 2GS and Lissamine Fast Red BG.

### Icyl Blue 2RS

This is the latest addition to the British Dyestuffs Corporation's range of Icyl (direct-viscose) colours for viscose artificial silk. Icyl Blue 2RS gives bright reddish blue shades of good fastness to water and washing. It possesses very good affinity for viscose artificial silk and gives even dyeings on material of irregular quality. The colour is suitable for the dyeing of cotton-viscose union material. Being easily dischargeable to a good white by either the Rongalite or tin process, Icyl Blue 2RS is recommended to the notice of printers for the production of dischargeable ground shades.

### Structure of Artificial Silk

The contribution by Mr. A. J. Hall, B.Sc., F.I.C., F.T.I., on "The fine structure of artificial silks in relation to dyeing and finishing," presented this week to the Manchester Section of the Society of Dyers and Colourists, was one of great scientific and practical interest. As Mr. Hall pointed out, in the dyeing and finishing of artificial silk, particularly fabrics, textile technologists frequently encounter subtle differences of behaviour between these newer fibres and those of cotton, and the resulting technical difficulties arising from these differences are often so considerable that it is important to ascertain by research the relationship between the fine structure of a fibre and its behaviour in technical processes. As a result of investigations into the behaviour of artificial silks towards moisture, dyes and particularly stretching, the following theory indicating the relationship between fine structure and physical and chemical properties has been evolved. The theory is apparently one which explains a number of existing facts observed in the dyeing and finishing of artificial silks and is therefore a useful one, but no more than this is claimed for it.

### The Fibres

It is assumed, and there is much scientific evidence for this assumption, that artificial silk fibres consist of innumerable small units or micelles of cellulose or cellulose ester, characterised by being much greater in length than in diameter. These micelles attract each other most strongly when in a position of end to end, and the properties of a fibre are largely dependent on the arrangement of the micelles of which it consists. The arrangement of the micelles within an artificial silk fibre are largely determined by the conditions of spinning (size of jet, rate of coagulation, and degree of stretching during coagulation), but may be modified by subsequent treatments such as treatment with swelling agents (*e.g.*, caustic alkalis) and stretching

whilst in a dry or moist condition. Similar results apply to viscose films.

Artificial silk fibres in which the longitudinal axes of the micelles are more parallel to the length of the fibre are less extensible, have greater tensile strength and lustre, and a diminished affinity for dyes and moisture. This condition is promoted by stretching. Fibres in which the micelles are "higgledy-piggledy" have greater extensibility, less tensile strength and lustre, but a greater affinity for dyes and moisture.

### Stretching

In technical textile processes stretching is a very important factor, since it plays a part wherever the textile material is handled. Stretching occurs in such processes as pining, winding, weaving, knitting, dyeing, and finishing. Hence the great necessity for its control within reasonable limits. (By reference to various dyeing and finishing processes, Mr. Hall was able to demonstrate the far reaching effects of stretching and to indicate the connection between these effects and the micelle structure as affected by stretching.)

Finishing processes invariably use stretching as a means for bringing artificial silk fabrics to their desired width and appearance. By reference to the results of investigations on the relation between stretching and softness and resistance to creasing, Mr. Hall was able to show that stretching easily decreases the softness of artificial silks and also renders them much more susceptible to creasing. This is, of course, a serious defect of finishing processes and one not hitherto recognised. Viscose silk is more creasable than cellulose acetate silk.

The facts disclosed in this interesting paper indicate the great need for co-operation between yarn and fabric manufacturers and bleacher, dyer and finisher for the production of artificial silk materials having the most attractive properties.

### The History of Scottish Dyes

In an article which appeared under the above title, in the November number of this supplement, the following statement was made in regard to the Grangemouth works of Scottish Dyes, Ltd.: "The works were designed by the late Sir Robert Lorrimer, the Scottish architect, who designed the Scottish National War Memorial in Edinburgh Castle." We understand that the reference should have been to the offices instead of the works. The works were designed by the company's own engineering staff, under the direction of Mr. G. W. Shearer, who has been in charge of the engineering section of Scottish Dyes since 1918.

### Change of Tone

The change in tone of Naphthol AS dyeings under the influence of wet and of dry heat has given occasion to several papers published in recent years, principally in *Melliand*. One of the most recent is that of Schwen (*Melliand*, 9, 673, 1928), who studied the question under conditions implied in the title, "Naphthol AS Studies on Cellophane." Dr. Kurt Scholl has examined the question on the basis of the solid azo-compounds themselves, with careful determination of the melting points and analytical determination of the carbon and hydrogen content. He decides that the change in tone of the derivatives studied is due to purely physical reasons, through aggregation of the finer particles of the lakes, that this aggregation takes place through the influence of water as an intermediary, even though water is present only in traces, and that there is no evidence for the view that the change is due to the taking on of water of crystallisation by the lake.

Dr. Scholl further concludes (and all this is concluded from actual chemical and optical study of the compounds) that the alteration of tone of an altered dyeing, such as is accomplished by very hot ironing of a sample which has

suffered alteration from the original dyed tone, is to be ascribed to sublimation of the coarser particles generated by aggregation, and the formation of an again finely divided deposit, the original tone being thus approached or even actually reattained. He adds that the situation of the particles of the lake within the fibre appears to have no influence upon the alteration of tone, and that (as one might expect) the fastness to light is improved through treatment of the goods wet and heat and decreased through the action of dry heat. This alteration in fastness can be clearly determined not only in cotton dyeings, but also in those on artificial silk, natural silk, and wool.

### A French Degree for Dr. Mullin

The University of Nancy, France, acting for the French Government, has conferred the degree of Doctor of Science on Professor Charles E. Mullin, of the Textile Chemistry, Rayon and Dyeing Division of Clemson College, South Carolina. Professor Mullin has been engaged for some time in research on the dyeing of rayon, cotton and other fibres and on the application of hydrogen ion control in textile processing. His theses at Nancy were along these lines. At Clemson he hopes to incorporate into the courses the mass of technical data on dyeing and rayon which he has collected during his recent visits to the textile centres of Europe. Professor A. R. Macormac, of Clemson, has spent the summer studying the textile industry and related subjects in New York, Philadelphia, and New England, giving special attention to the technical analysis of textile chemicals, microscopy, and laboratory work.

### The Study of Perspiration Effects

In research on the fastness of dyes, perspiration with its effects on colour and finish, remains one of the problems. Perspiration is an agency difficult to study because it cannot be easily controlled. Even sunlight is so nearly mechanical in action over a given period of seasons that laws of fading can be established, involving even such variable factors as humidity. Washing is obviously a combination, basically, of mechanics and soap chemistry, and testing methods for use with the Launder-Ometer or other laboratory washing machine now present very few problems. But perspiration must be recognised as a peculiarly *personal* colour-destroying agency. It may vary almost to the extent that human character varies. There can, therefore, hardly be a standard for perspiration. A dozen standard types may be necessary. The factor of time enters to complicate tests, for the chemical constitution of perspiration will change on standing.

"The ammonia-nitrogen content was found to increase on standing," states a recent comment in *Textile Maintenance Notes*, a bulletin issued by the Textile Division of the U.S.A. Department of Commerce. "This probably explains why some investigators found perspiration alkaline. Their examination was probably made some time after the perspiration was given off, with the result that it analysed alkaline. The rapid rise in ammonia content of perspiration was found to be due to the presence of an activator bacterium normally present on the skin. This leads to the conclusion that in any problem regarding the fastness of dyes to perspiration, regard must be paid to the changes of the constituents of perspiration due to the bacterial infection of any garments worn near the skin. Much of the importance of these experiments lies in the fact that they may permit the development of a synthetic perspiration by means of which dyes and fabrics may be tested before being marketed."

Despite the tests for fastness to perspiration that may now be in use, it is evident from this opinion that the subject calls for further study. Some may consider perspiration as a minor matter in fastness research, but it may yet be disclosed as a problem more difficult than the standardisation of light fading.

## **The Fastness of Lake Colours to Light**

**By S. T. Kinsman**

*A paper under the title "Notes on the Fastness to Light of Lake Colours" was recently read by Mr. S. T. Kinsman, A.M.C.T., before the Oil and Colour Chemists' Association, and is reprinted below from the Association's Journal.*

SOME three years ago the author of this paper was a member of the Sub-Committee of the Colour Makers' Association appointed to carry out a investigation on the fastness to light of lake colours. The matter contained in this paper has been prompted by this circumstance. During the period of the experiments it became more and more evident that the study of the property in question had been badly neglected, and some extraordinary results were obtained from certain colours. In the past, the fastness of a large number of lakes has been taken for granted, the classification being based on the fastness of the dyestuff from which they were derived; whole groups of dyestuffs being grouped together as being of similar fastness to light.

### **Lake-Makers' Difficulties**

The question is sometimes put: "How is it that lake-makers are unable to guarantee their products in the same way that textile dyers are able to do?" The chief reason is that lake makers do not know how their colour is going to be used. Another reason is that, generally speaking, dyestuffs are faster to light on the fibre than they are in the form of a lake. There is, however, still another reason, perhaps not very often realised and that is, that there are few dyestuffs which are guaranteed by the textile user which are of use to the lake-maker. The vat colours are referred to particularly; they have a very limited use for lakes, and these form the bulk of the textile colours which can be guaranteed. Another question which is often asked is: "Well, how long will the colour stand?"—and this paper endeavours to show that it is impossible to answer this question accurately. Only comparisons can be given.

If a standard method for testing these lakes could be set up, even if only tentatively, it should help to introduce eventually some order into the tangle which undoubtedly exists at present. This paper, therefore, consists of a brief survey of the factors governing the fastness to light of lake colours and the description of a series of experiments carried out with the object of defining standards of light fastness and the manner of testing by natural and artificial means, particularly the latter.

### **Classification**

At the present time there are no recognised standards of light fastness of lakes in this country, whilst there is at least an arbitrary scheme in use on the Continent. Such terms as are used, namely, Excellent, Very Good, Good, Fair, Poor, mean very little except to the person employing them. It seems expedient that some more definite scheme be put into use; so that this paper really resolves itself into an appeal for classification.

The subject is dealt with under the following heads:—

Course and cause of fading; effect of different media; daylight tests; lamp tests; attempted correlation between daylight and lamp; and suggested system of classification.

All lakes change under the influence of daylight, whether in the dry form or when rubbed out in a medium. In so far as the changes originate through the medium as the main cause, they lie outside the scope of this paper. Much trouble is caused through confusing the light fastness of a colour with its fastness or otherwise to chemical action. In considering the probable durability of a lake the effect of various reagents should be taken as separate factors. We shall see later that the effect of the medium, even where it exerts no chemical influence, should be taken as a separate factor in reference to durability.

### **Optical Changes**

The optical changes which take place when a lake fades can be said to proceed in two main ways. The change is usually evident along all sides of the colour triangle, the fading proceeding until a point of greyness is reached. In other cases, a darkening takes place at the beginning, and disappears towards the end of the fading. These may be looked upon as direct and indirect courses of fading. As the fading and darkening proceed, there occur changes in the degree of selective light absorption, which later discontinue in favour of increased reflection or absorption.

The cause of the change is due to light of all wave-lengths of the visible and the invisible spectra, for, although ultra-violet light in general produces an especially powerful effect, the change due to light is not limited to the effect of the rays of short wave-lengths, for there are some colours which change more slowly in ultra-violet light than they do in sunlight. Examples of this are seen in certain acid-green lakes.

The reason for the fading of lake colours also lies outside the scope of this paper, but, generally speaking, it is due to the decomposition of the dyestuff constituent by oxidation or reduction or both. Generally speaking also, the more chemically stable the salt of a dyestuff is, the faster to light the lake will be; in its particular class of course. For example, all the latest work on the improvement in the fastness to light of basic and acid dyestuffs depends on this fact. Basic and acid dyes become faster to light by the formation of stable complex combinations. Acid dyes which contain amino groups become likewise faster to light when not only the sulphonie acid groups but also the amino groups are fixed in laking. The alkali salts of the almost insoluble pigment dyestuffs of the Lithol Red type are less stable than the barium and calcium salts.

### **Fastness of Lakes v. Dyestuffs**

In stating the fastness of a lake it is far too common practice to give it the fastness of the dyestuff from which it is produced. This is particularly bad, and every lake should be tested on its merits before anything is said on this particular property. It is impossible to say anything definite on the effect of various extenders on the fastness of a lake but, generally speaking, and apart from any chemical effect the extender may exert, it may be said that there is a falling away in fastness to light of the exposed colour with increase in the amount of extender. What exactly is meant by the "exposed" colour will be seen presently. It is noteworthy that Professor F. M. Rowe, speaking of the fastness to light of azo colours on the fibre, suggested that increased fastness to light was due merely to decrease in the surface area of the colouring matter exposed, and the same can be said of most lake colours. This brings in the feature of dispersion, so that altogether the system is rather complicated.

The effect of various media on a lake is of great importance. It is often said that a colour is faster in one medium than in another. Apart from any chemical effect, this discrepancy in fastness is only apparent and usually due to the difference in the actual amount of colour it is possible to expose in such a medium. To explain: there are two main types of effect due to the medium—a surface effect and a depth effect.

### **Surface and Depth Effects**

When a water paint or distemper colour is faded, the colour is destroyed on the surface only, and by carefully rubbing off this faded layer the original colour can be restored, and with the usual distemper coating this operation can be repeated several times so that the fading is only superficial. This is known as a surface effect.

On the other hand, if a lake is rubbed out in varnish and exposed, the effective light will penetrate more deeply into the pigment, so that more than the surface layer is faded. This is known as a depth or optical bridge effect, and with increase in the difference of refraction the influence of the medium increases, and there will be an increase in the fastness to light with increase in the thickness of the filtering layer.

There is still another part played by a medium, i.e., in the superficial coating of a lake with a medium such as varnish. When light passes through a transparent medium of low refractive index to one with a high value, part of the light is transmitted and part reflected, and, generally speaking, the greater the difference between the refractive indices of the two media the more light will be refracted at the interfaces and the less transmitted, or—in the case of pigments—absorbed. This effect of coating with a clear medium can be looked upon as subsidiary to the depth effect—which it increases. The highest refractive index is that of an oil

medium, oil and varnish emulsions coming next, then dextrin, casein, animal glue and, lastly, vegetable gum.

It will be seen that if one could succeed in producing a layer of such thinness as not to exceed the depth of the light effect on the colour then one and the same colour would prove to be of similar fastness to light in all media, and it is on such a basis that lakes should be classified. The effect of various media, thickness of film and so on, could then be taken as separate factors, bearing in mind that the increase in fastness to light is limited to the thickness of the layer through which the light is able to penetrate.

#### Fading Experiments

Let us consider a number of experimental rubbings. These rubbings are produced from the same lake, part of which has been rubbed out in glue size water medium, and the other in lithographic varnish. They were exposed in a fading lamp, simultaneously, for 4, 8, 16 and 32 hours respectively. The glue rubbing demonstrates very clearly the surface effect, careful scraping showing unfaded colour underneath. The lithographic rubbing may be taken as an example of the depth effect. It may appear contradictory to find that the glue rubbing is faster than the lithographic, but it must be borne in mind that, whereas a coated paper of this description contains on the average 5 grms. of colour per sq. metre, the printed paper probably contains nearer 0.5 gm. Although, in the case of the coated paper, the colour may be faded on the surface, the unfaded colour still shows through and masks the fading effect; in other words, it is not a layer of the ideal thickness referred to previously.

Dr. S. G. Barker, speaking in relation to wool dyeing, pointed out that it has been proved mathematically that, for a large number of colours, the amount of fading is approximately proportional to the square root of the time of exposure, although in the earlier stages of fading the loss of colour is approximately proportional to the time. This is undoubtedly analogous to the course of the fading of some lakes, e.g., the lithographic rubbing considered above, but in many instances practically the reverse is the case, where fair thicknesses of transparent film are concerned. In such examples we must assume that destruction of the colour *does* take place in the initial stages, but that it is not very perceptible. Actually, however, there is no definite course in the fading of lakes. The course described by Dr. Barker refers to the change in the degree of selective light absorption and reflection which takes place as the fading proceeds.

#### Standard Methods of Testing

There is no doubt that standards of fastness to light can only be determined on a graphical basis, but it first becomes necessary to create standard methods of testing, and from these a practical classification can be made. To this end the following experiments were carried out.

In practice the thinnest layer of colour is the lithographic print, bearing in mind the layer of ideal thinness. First of all, therefore, a series of 10 per cent. lakes was prepared from practically every known type of dyestuff, each one ground in lithographic varnish and printed on neutral glazed paper. These prints were placed in a wooden cabinet of the following description:—

*Base.*—2 ft. 3 in. square.

*Depth.*—1 ft. at top, 4 in. at bottom.

*Glass.*—2 ft. square  $\frac{1}{8}$  in. thick "vita" glass, sloping at 30° to the horizontal.

*Exposure Board.*—Of similar size supported with face  $\frac{1}{2}$  in. from the glass.

*Air Holes.*— $\frac{3}{8}$  in. in diameter. About 40 in number are placed round the edge of the board and in the sides of the cabinet to allow for the free passage of air over the surface of the exposures. The glass was placed at 30° to the horizontal so that the angle of incidence of the sun's rays would be as near as possible a right angle in the summer months.

The cabinet was placed in an exposed position facing south, a sunlight recorder being installed alongside. The colours were separated into two classes, namely, those which are known to be more fugitive, and those which are more permanent. The fugitive class was exposed for the first week in each month of April to August inclusive, a different portion of the print being exposed each time.

The more permanent rubbings were dealt with in three lots as follows: (1) The effect of each month's exposure April to August being noted separately; (2) the cumulative effect

produced over the same period; (3) the effect of exposing the prints for the three summer months June, July and August. The number of hours of sunshine during these various periods was noted on the recorder.

Before proceeding further we must consider how the results are to be classified. There are several methods in use. Sometimes 10 classes are employed, No. 1 being the most permanent, or the reverse method may be adopted, i.e., with No. 10 the most permanent. The consensus of opinion, however, is that five classes are sufficient, No. 1 being taken as the most permanent, i.e., first class fastness. Incidentally, this classification is employed by textile users of dyes, and, generally speaking, it is the method in use on the Continent.

The five classes, therefore, we shall define for the present as follows: (1) No perceptible alteration after one month's exposure—June, July or August; (2) a slight loss in depth or alteration in shade after one month's exposure; (3) a distinct loss in depth or alteration of shade after one month's exposure; (4) a distinct loss in depth or alteration of shade after 14 days' exposure; (5) colour practically bleached out after one week's exposure.

#### Results

The first obvious result of exposing the lakes as described was that not one remained absolutely unaltered after three months. The next observation is that the number of hours sunshine from one month to the next does not govern the amount of fading produced, the actinic value of the light in any particular month appearing to be the factor involved. For instance, the fading effect of December sunshine is very small indeed. In this connection it is interesting to note that Dr. Ewart Moss and Mr. Arthur W. Knapp have evolved a chemical method for the standardisation of ultra-violet light by means of an instrument called the Uroxameter, that gives the values of light as a definite chemical factor, from its action on a solution of uranium acetate and oxalic acid.

Patterns of the colours in question were exposed in various parts of the country under the same conditions, and the results were practically the same in every case, so that the colours were easily placed in the five classes. These were carried out by different observers, and this demonstrates that if a specified method of testing were adopted there would be little difficulty in classification.

In these days of speed, however, one cannot wait a month, or for the summer months, to ascertain the comparative fastness of a lake, so that an attempt must be made to reproduce the fading of these colours by artificial light. It is obvious that if a lake could be exposed under the same conditions under which it would be used there would be no difficulty in classification. A few experiments with a mercury vapour lamp confirmed the idea that this apparatus was unsuitable for the purpose. With the carbon arc, however, the spectral curve is more comparable with that of sunlight, and on this account alone is more likely to give consistent results.

The artificial fading of the prints was therefore carried out by means of a carbon arc lamp. Portions of the same rubbings, which had been exposed to daylight, were then exposed in the lamp, and an attempt made to match the fading produced by daylight during July, 1927. The results obtained were on the whole rather disappointing, and no direct equivalent to daylight fading could be obtained. A rough figure gave 64 hours' exposure as being equivalent to one month's summer daylight exposure. [After describing various results, Mr. Kinsman states that endless difficulties stand in the way of correlating results between daylight fading and artificial fading.]

#### Method of Comparison

After carrying out the daylight exposures, a clear classification was effected, and there was complete unanimity among the various observers. The idea brought out—of course not new—is that it should be possible to take a lake representing each of the five classes, and compare all lakes against these standards.

It is suggested that the following lakes be taken to represent the five classes of fastness to light:

- Class 1. Alizarine Red Al Ca phosphate lake.
- " 2. Permanent Red 4B Ca Salt.
- " 3. Lithol Red R Ba Salt.
- " 4. Eosin Y.S. Pb Salt. BaSO<sub>4</sub> base.
- " 5. Fastness less than that of Eosin.

It is to be particularly noted that each member of the five classes is the lowest of its class. Any lake under examination which does not attain the fastness of the colours described automatically falls into a lower class. Lakes of the dyestuffs in question, alizarine red, permanent red 4B, lithol red R, and Eosin Y.S. were taken, and rubbings made in the manner described, i.e., a film, as thin as possible, of pigment in litho varnish, was applied to the paper and exposed in a lamp of another type, and observations made by an independent investigator at the Federation Research Association.

A description of the lamp and method used, in this instance, is as follows: The samples were tested, at an average temperature of 58° C., in a circulating air stream, the relative humidity being 75 per cent. The lamp consisted of a plain carbon arc, enclosed in a glass globe of such a composition as to transmit some of the ultra-violet light. The samples were carried in metal boxes with "vita" glass windows, which also transmitted some of the ultra-violet light. The distance from the arc to the specimens was 11½ inches, and the current passing through was 12.5 amperes D.C. The constant humidity feature of this lamp is an important one, as fading of colours is greatly dependent on moisture, in fact, some colours, which normally are quite fugitive, will not fade at all when exposed in vacuum, or a perfectly dry atmosphere.

#### Fifty Hours' Exposure

Patterns exposed up to 50 hours showed that the lake representing Class 1 was practically unaffected, Class 2 definitely faded, Class 3 bleached out after 47 hours, and Class 4 bleached out after 27 hours. To show that such a method allowed for a fair margin of error, rubbings were taken of two thicknesses of a pure lithol red lake, and it was found that both were bleached out in the same time.

Further experiments, with the first lamp described, and the same rubbings, gave:

- Class 1. Unbleached after 240 hours.
- " 2. Practically bleached after 120 hours.
- " 3. Bleached after 60 hours.
- " 4. Bleached after 34 hours.

Further exposure produced no effect.

It will be seen that the lakes described are of great use in standardising the various fading lamps at present in use. One point of importance in employing artificial fading lamps is that prolonged exposures should always be carried out, as the course of the fading varies tremendously.

If a lake of No. 2 fastness, for example, is practically faded out in 120 hours by this method, it cannot be said that it will fade completely in approximately two months English summer daylight exposure. If it is used to produce a tint on a poster, then the comparison is correct, but, as a high class enamel, it will last perhaps 20 times as long.

It must be admitted that in testing lakes, fading induced by artificial means can only serve as a guide to the actual fastness, but that, if the tests are carried out in the manner described, there will be comparatively few anomalies. Experience is required with these lamps; but lack of such experience should not stand in the way of classifying lakes under the scheme described. Users could make themselves familiar with the behaviour of the lakes mentioned, and know what to expect from colours compared with them. The method is not put forward as giving absolute values for fastness; it is entirely comparative, and one which I hope is, at least, practical.

#### New Bleaching Product and Process

PATENTS were recently granted to United Chemical Products, Co., of Jersey City, N.J., on a product and process developed in its laboratory for kier boiling and bleaching in one operation. According to the statement of the manufacturer, this invention relates to an improved and simplified process for cleaning, scouring, bleaching and colour-fixing cotton raw stock, yarns, warps, piece goods, linen, wool, silk and other fibrous and textile materials. The patents also cover a bleaching composition known as Bleachall for use in practising the process.

One of the objects of this new process, it is further stated, is to simplify the known treating processes, to minimise the multiplicity of steps and to provide a process which can be practised as a single step even in iron kiers and without any deleterious or objectionable effects on the resulting goods.

The invention particularly features what is described as

an improved process constituting a finish for woven fabrics, such as Turkish towels, which include both white areas to be bleached and coloured areas or bands to be rendered brilliant and in clear contrast to the white area. According to the description, this phase of the process is attained by subjecting the goods while in an iron kier, if desired, to a single boiling treatment, and in the presence of reagents capable of effecting the desired bleaching and colour setting effect, but incapable of forming iron rust with the kier. There takes place a combined reducing and oxidizing reaction, it is claimed, capable of bleaching and setting colours, and at the same time, composed to form incidentally a coating to prevent the formation of rust with the iron kier.

#### Belgian Dye Market

BELGIUM is not a producer of dyes derived from coal tar, and imports originate in Germany, France, Switzerland, and the United States. Approximately 80 per cent. of the total import is consumed in the textile industry. Although the recent increase in the production of rayon in Belgium has afforded a slight additional demand for imported dyes, consumption, on the whole, has remained practically stationary during the past two years as a result of the quiescent condition of the cotton and wool industries. The current situation of the Belgian dye market has been improved considerably by the recent combination of German, French, and Swiss enterprises. Previously the Belgian market for dyestuffs comprised a large number of small independent concerns whose primary selling policy was price cutting. This condition became so acute that Swiss agents were forced out of the market because of their inability to compete with the reduced quotations offered by the stronger American and German manufacturers. Since the Swiss interests have become affiliated with the I.G. and the French group, however, their position has been materially strengthened, with the result that Swiss dye products are now entering the country. This situation applies particularly to various colours in which Swiss manufacturers specialise.

Another favourable factor affecting the present market is the termination of the regulation of the dye trade by the reparations agreement. It is reported that formerly reparation dyes were resold frequently at a considerable discount to the Belgian agents of German manufacturers, and that these agents accordingly, were able to offer dyestuffs at extremely low prices, thus affording almost insurmountable competition. Fortunately, this element has been entirely eliminated, and, furthermore, it is reported that stocks of dyes held by the Belgian Government as payment in kind have been entirely liquidated at the present date, with the result that the position of American dyes in the Belgian market has been greatly strengthened. In 1928 approximately \$250,000 worth was exported from the United States to Belgium.

#### United States Dye Exports Increase

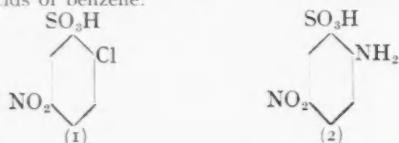
SHIPMENTS of coal tar, colours, dyes, and stains (exclusive of package dyes, for household use) totalled, during the first six months of 1929, \$3,699,186—an increase of nearly 33 per cent. over shipments during the corresponding period of 1928, when \$2,796,749 worth were exported. Values of shipments to the principal countries during the period, January-June, inclusive, during 1928 and 1929, are given below:—

Country.	First half 1928.		First half 1929	
	Value. \$		Value. \$	
Belgium .. .. .	108,965	..	134,220	..
Czechoslovakia .. .. .	1,968	..	12,735	..
Germany .. .. .	43,148	..	132,476	..
Canada .. .. .	520,808	..	486,775	..
Mexico .. .. .	52,022	..	37,105	..
Cuba .. .. .	48,165	..	14,919	..
Argentina .. .. .	36,181	..	63,017	..
Brazil .. .. .	61,488	..	20,254	..
India .. .. .	355,071	..	273,206	..
China .. .. .	974,350	..	1,784,854	..
Hong Kong .. .. .	36,225	..	84,693	..
Japan .. .. .	464,049	..	540,638	..
Philippines .. .. .	21,221	..	50,051	..

## Basic Intermediates for Dyestuffs: No. XXXII.—Some Sulphonic Derivatives of Benzene

By "Consultant"

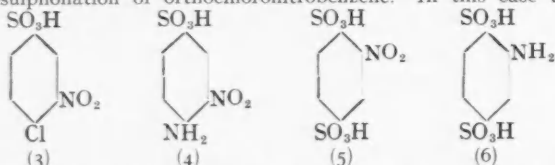
THE important function of the sulphonic acid group in the solubilising of dyestuffs makes the intermediates containing such a group of great importance; thus, the simple sulphonic acids of aniline, sulphanilic and metanilic acids, are without doubt in the first rank of intermediates for azo dyes. In this contribution it is proposed to deal with the substituted sulphonic acids of benzene.



One of the parent intermediates of this group is 2-chloro-5-nitrobenzenesulphonic acid (1), which can be converted into the corresponding nitro amino compound. 2-Chloro-5-nitrobenzenesulphonic acid is obtained by the direct sulphonation of paranitrochlorobenzene. It will be observed that the ortho-para directing influence of the Cl group is assisted by the meta directing influence of the nitro group, so that only one isomer is formed during the sulphonation. The method used is as follows: The chloronitrobenzene (1 cwt.) is melted and allowed to flow with stirring into 20 per cent. oleum (1½ cwt.) at 100° C., and as soon as the addition is complete the temperature is raised to 160° C., where it is maintained for six hours. When the mixture has cooled to room temperature it is run out on to crushed ice (3 cwt.). On standing crystals separate out, which are filtered off, or centrifuged from the acid liquor, and dissolved in water. The residual chloronitrobenzene is removed by blowing steam through, and the acid required is salted out from the cooled liquor by the addition of hydrochloric acid. The yield of 2-chloro-5-nitrobenzenesulphonic acid is exceptionally good, approximating to 90 per cent. of the theoretical.

As with many of the chlorinated aromatic derivatives containing an acid substituent, the chlorine in 2-chloro-5-nitrobenzenesulphonic acid can be replaced by the NH<sub>2</sub> group by the simple expedient of treatment with ammonia. Autoclaving with an alcoholic solution of ammonia for 2-3 hours at a temperature of 120-140° C. is capable of effecting this transformation, but a better method for works practice is to stir the press cake of 2-chloro-5-nitrobenzenesulphonic acid with an equal weight of ammonia (S.G. 0.880) and to autoclave the solution at 150° C. for eight hours. On cooling the liquid from the autoclave, the ammonium salt of the 2-amino-5-nitrobenzenesulphonic acid (2) separates in the form of large amber-coloured cubes, which, if necessary, may be converted to the parent acid by treatment with the theoretical quantity of hydrochloric acid.

The preparation of the isomeric 4-chloro-3-nitrobenzenesulphonic acid (3) requires a slightly different treatment, although the method is essentially the same, namely the sulphonation of orthochloronitrobenzene. In this case the



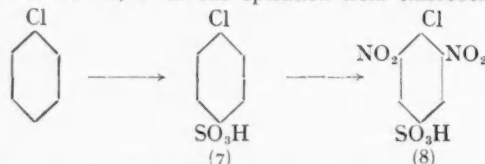
orthochloronitrobenzene is allowed to run into five times its weight of 30 per cent. oleum, and the sulphonation is completed by stirring for some hours at 100° C. The temperature is lower than that used during the preparation of the previous isomer, in order to reduce the amount of the other isomers formed (notably 2-chloro-3-nitrobenzenesulphonic acid). To bring about the sulphonation in reasonable time the amount of SO<sub>3</sub> in the oleum is increased to 30 per cent. After the sulphonation is complete, the acid is isolated by pouring on to ice, neutralising the liquid with lime, filtering off the clear solution of calcium salt, and treating it with sodium carbonate to obtain the sodium salt. The solution of the latter is filtered from calcium carbonate, and treated with hydro-

chloric acid to liberate and salt out the free 4-chloro-3-nitrobenzenesulphonic acid. The free acid crystallises with one molecule of water of crystallisation, and may readily be converted into the corresponding amino compound (4) by the process just described in connection with the corresponding 2-chloro-5-nitro compound. The two processes are so similar that no details need be appended.

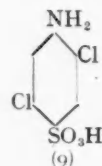
An additional interest attaches to 4-chloro-3-nitrobenzenesulphonic acid, in that it is the source from which aniline-2:5-disulphonic acid is obtained. When the sodium salt of the chloronitro acid is treated with sodium sulphite the chlorine atom is replaced by the SO<sub>3</sub>H group, giving the 2:5 disulphonic acid of nitrobenzene (5), which may then be reduced to the corresponding derivative of aniline (6).

To effect this transformation, the sodium derivative of 4-chloro-3-nitrobenzenesulphonic acid (1 cwt.) is dissolved in water (2½ cwt.) and the solution boiled with crystalline sodium sulphite (1½ cwt.) for two hours. A reflux condenser is used to keep the amount of water constant. On cooling the nitrobenzene-2:5-disulphonic acid separates out in long crystals, and a further crop of the latter may be obtained by salting out. The nitro compound is pressed as dry as possible in the filter press, and reduced without delay. The press-cake of nitro compound (1 cwt.) is dissolved in six times its weight of water, and the reduction effected by the gradual addition of iron dust (1 cwt.) and 30 per cent. acetic acid (40 lb.). Stirring is maintained at 100° C. until the conversion to amino compound is complete. The whole is then made alkaline and filtered. The solution, after concentration and acidification, gives the acid sodium salt of aniline-2:5-disulphonic acid (5). It is also possible to obtain, from the latter compound, the corresponding phenolic compound by the replacement of one of the sulphonic groups by an hydroxy group. The compound so obtained is not of great importance.

A further intermediate which is derived from 4-chloro-3-nitrobenzenesulphonic acid is 4-chloro-3:5-dinitrobenzenesulphonic acid (8), obtainable by the further nitration of the parent derivative, or in one operation from chlorobenzene.



In the former case the 4-chloro-3-nitrobenzenesulphonic acid is converted to its potassium salt and dissolved in an equal weight of 25 per cent. oleum. The theoretical amount of nitric acid is slowly run in, and the progress of nitration is assisted by heating to 120-130° C. for a time. The dinitro-compound is isolated by pouring into water. The preparation in one operation from chlorobenzene is, however, by far the best method for the preparation of this intermediate. The chlorobenzene (1 cwt.) is gradually run into a mixture of sulphuric acid monohydrate (240 lb.) with 25 per cent. oleum (100 lb.) and the whole stirred at 100° C. until all the chlorobenzene has gone into solution. The nitric acid is added (86 lb. of 87 per cent.) after the reaction mixture has been allowed to cool, and at such a rate that the temperature does not rise above 40° C. After standing two hours, 60 per cent. oleum (3 cwt.) is added, together with potassium nitrate (1½ cwt.). The temperature is maintained at 120-130° C. for 2-3 hours. The product is easily isolated by pouring into water. It is capable of reduction to the diamino compound, which finds some use as a middle component in the preparation of azo dyes.



The only remaining intermediate of this series which is of any considerable importance is the sulphonic acid of 2:5-dichloroaniline. This may be obtained in a very simple manner. 2:5-Dichloroaniline is melted and allowed to flow

into three times its weight of 18 per cent. oleum. The mixture is heated to 170–180° C. for three hours, and poured on to ice after cooling. The acid (9) separates out and need only be filtered and washed.

## Dyes and Their Application: Recent Technical Progress

By L. J. Hooley

THE cellulose silks, being similar to cotton in chemical composition, behave similarly towards dyestuffs, that is, they can be dyed with the same broad groups of colours, namely, the direct azo, insoluble azo, basic, sulphur and vat classes. Where fastness is necessary the vats are used, but otherwise the direct colours are the most convenient.

### Even Dyeing of Cellulose Silks

In spite of this general similarity, when it comes to the choice of specific dyestuffs, the same colours are often not suitable for both cotton and artificial silk, because of differences in physical properties, and because the dyeing of artificial silk is a more delicate operation than the dyeing of cotton; so that in practice only a few of the cotton colours are found suitable for artificial silk. The alteration which the cellulose undergoes in the dissolving and regenerating processes increases its affinity for dyestuffs, which appear to be very sensitive to differences in physical properties, so that even the same thread may, in extreme cases, dye deeper in one place than another.

It was early recognised that different kinds of cellulose silks, e.g., the viscose, cuprammonium and nitrocellulose varieties, could not be used together, but some trouble was encountered before it was learnt that batches of, for example, viscose from different makers, or even different batches from the same makers, should not be mixed, since any alteration in the method of production is likely to show up visibly after dyeing. As indications of the slightness of variations in manufacture which will cause trouble, a recent paper of Lehner and Jaeger may be mentioned. These authors show (*Kunstseide*, 1927, 219, 264) that dyeing properties are affected by the tension of the thread during coagulation. Increase of tension makes the silk less receptive to direct and vat dyes and more to basic dyes, while other physical conditions during spinning also affect the properties of the material.

### Effect of Increased Affinity

The fact that these silks have excellent affinities for dyestuffs tends to accentuate rather than diminish the trouble, since quick dyeing is always liable to result in unevenness. Also, when artificial silks are spun in continuous lengths, the same mixing and averaging does not take place as in the case of the cotton and wool staple fibres. Weaving is again found to accentuate the unsatisfactory results, since the variation shows up as bars or other quasi-pattern effects.

While speaking of artificial silks, mercerised cotton may be mentioned, since for obvious reasons it behaves somewhat similarly and shows the same increased affinity and liability to unevenness.

As regards the technique of dyeing to overcome the above-mentioned difficulties, much depends on the correct choice of dyestuffs. Apart from indicating the products which are most suitable, manufacturers have played a positive role by synthesising special products. In this direction, much has been done by Imperial Chemical Industries, who have prepared a large number of new colours for dyeing regenerated cellulose silks, selected members of which have been placed on the market as the Icyl series. This now includes Icyl Blue Black 6BS, Blue GS, Brown GS, Orange GS and RS, Red GS and Violet BS. As regards new dyeing methods and selection of dyes, etc., Courtaulds have, of course, been largely responsible: some of the latest and most interesting of these will be briefly described.

### Selection of Dyestuffs

To begin with, Courtaulds have recommended a test for dyes. With each dyestuff eight dyeings are carried out at temperatures of 20°, 30°, 40°, 50°, 60°, 70°, 80° and 90° C., other conditions being identical. The temperature at which the deepest dyeing is obtained is noticed with each dyestuff. Those dyes should then be used together which agree as to temperature or which come nearest. As a matter of expe-

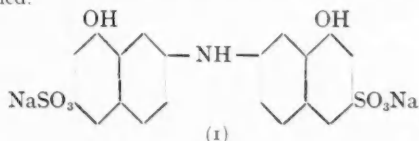
rience, it has been found that the colours which have maximum at 20° C. give level results most easily. (In parenthesis, it must be noted that a dye which shows a maximum at 20° C. does not necessarily dye best at this temperature, and will in general be used at 70° to 90° C.). Similar tests can be used for determining whether different batches of silk can be used together.

### Dyeing Methods

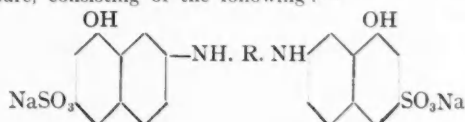
It has recently been found that improved results can be obtained by dyeing in the presence of a suitable inorganic salt with  $\alpha$ - or  $\beta$ -naphthol (B.P. 314,761: Courtaulds, C. M. Whittaker and T. M. Harrison). An example describes the dyeing of 100 lb. of yarn in 400 gallons of water containing 1 lb. of colour, with 1 per cent. each of  $\beta$ -naphthol and salt, at 90° C. for half-an-hour. In this bath, different batches can be dyed together or batches which have emerged unlevel can be retreated.

According to Alterhoff (*Textilber.*, 1927, 758) the material can be first scoured in a bath containing 0.05 per cent. of Monopol soap and 0.1 per cent. of soda ash. This is followed by entering into a boiling liquor containing 20 per cent. of Glauber's salt and a little soluble oil. After a quarter of an hour's treatment in this second bath, the dye is added and dyeing continued for three-quarters of an hour, this being followed by a cooling bath.

Returning to the work of Imperial Chemical Industries, a selection from the large number of patents may be mentioned. In B.P. 270,446 (B.D.C., J. Baddiley, P. Chorley and R. Brightman), diazo dyes are made from 8:8-dihydroxy-2:2-dinaphthylamine-6:6-disulphonic acid (1) or the corresponding 3:6:3':6'-tetrasulphonic acid by coupling with diazo compounds. Thus from one molecule of the disulphonic acid and two molecules of *p*-amino-acetanilide a violet black is obtained.



Somewhat similar compounds are used in B.P. 270,883, except that the principal component is slightly different in structure, consisting of the following:—



where R is either a carbonyl, thiocarbonyl or straight chain of methylene or carbonyl groups. By coupling this with a large number of amines and their sulphonic acid, reds, violets and browns can be obtained.

In B.P. 296,011 (B.D.C., R. Brightman, and P. Chorley) tetrazotised 4:4-diaminodiphenylmono-, di- or trisulphide is coupled with two molecules of 2-amino-8-naphthol sulphonic acid; alternatively one of the molecules of the acid may be replaced with another component. 4:4-Diaminodiphenylsulphide with salicylic acid and phenyl gamma acid gives a brown. Somewhat similar is B.P. 296,047.

Instead of 2-amino-8-naphthol sulphonic acid, 2-amino-5-naphthol-7-sulphonic acid (J acid) may be used, or its nitrogen substituted derivatives, monazo dyes dyeing in level shades being obtained by coupling with diazotised amines or diazotised amino-sulphonic or carboxylic acids. As an example, the glycine derivative is coupled with diazotised aniline (B.P. 280,320 B.D.C. and J. Baddiley).

The other patents which have not been included are by the same inventors as those mentioned above.

### The Italian Dyestuff Industry

#### An Account of its Development

It is only since the war that Italy has become a producer of synthetic coal-tar dyestuffs. The existence of large plants for the manufacture of high explosives required during the war, and the problem of their utilisation in time of peace, led to the establishment of the Italian dyestuffs industry, and the end of hostilities witnessed the first steps in the manufacture of such leading intermediate products, obtained from aromatic hydrocarbons, as benzol, phenol, toluol, xylol, naphthalene and anthracene, from which the whole intricate scale of colours required by the modern dye industry is now obtained.

#### Raw Materials

Italy imports part of the raw material required, and secures the remainder by the scientific treatment of the by-products of the distillation of imported coal from her gas and coke industries. From the distillation of coal oils and the washing of gases from coke ovens the Italians have secured of recent years ever-growing quantities of light oils (3,843 metric tons in 1926, 4,743 in 1927), from which 1,483 tons of rectified benzol were distilled in 1926, and 1,390 tons in 1927; 1,950 tons of naphthalene in 1926 and 1,800 in 1927; 175 tons of phenol in 1926 and 304 in 1927, etc. The supply from these sources is, however, still inadequate to meet the home demand.

The leading intermediate products now manufactured in Italy are aniline oils, of which 11 million kilograms are now produced annually, and a yet larger quantity of chlorobenzol, in the production of which part of the chlorine obtained in the manufacture of caustic soda by the electrolytic process is utilised. Dinitro-chlorobenzol and dinitrophenol are obtained from this chlorobenzol, besides 350 metric tons of hydrochloric acid. Other manufactures include annually 250 tons of betanaphthol; 250 tons of benzidine; 150 tons of paranitroaniline; as well as tolidine, alphanaphthylamine, gamma acid, naphthionic and sulphanilic acids, etc.

#### Output

The most important Italian plants are those at Cengio (Savona), originally equipped for the manufacture of high explosives, and subsequently adapted to the distillation of intermediate products and aniline oils. Next in importance comes the factory at Cesano Maderno (Milan).

The following figures show total output:

	Aniline.	Hydrochloride of Aniline.	Intermediate Organic Products.
1921 . . . . . metric tons	184	—	920
1922 . . . . . "	403	—	1,708
1923 . . . . . "	480	2	2,450
1924 . . . . . "	900	65	2,370
1925 . . . . . "	1,030	74	4,990
1926 . . . . . "	930	132	5,290
1927 . . . . . "	680	214	4,500

The success obtained in the manufacture of intermediate products has naturally led to a similar development in the manufacture of dyestuffs, some of which are now produced in quantities considerably in excess of the requirements of the home market. The Italian output now covers most of the more important products for the whole series of synthetic dyes. The most notable results have, however, been obtained in the manufacture of sulphide colours, which account in the last few years for nearly 70 per cent. of the total output.

#### Synthetic Indigo

The manufacture of synthetic indigo, begun in 1926 at Cesano Maderno, and that of Navy Blue which followed in 1927 at Conca Fallata (Milan), were the first steps taken to manufacture vat dyes. The Cesano Maderno factory for synthetic indigo brings the total number of such plants in the world up to eight. The Italian output capacity stands around 6,000 kg. per day, as compared to an annual demand on the home market estimated at 600,000 kg. The output of other organic dyestuffs is shown in the following table:

	OUTPUT OF DYESTUFFS.		Total.
	Sulphide Dyes.	Others.	
		Kilograms.	
1921 . . . . .	2,887,000	700,000	3,593,000
1922 . . . . .	3,850,000	1,050,000	4,900,000
1923 . . . . .	3,705,000	1,815,000	5,580,000
1924 . . . . .	3,960,000	1,685,000	5,645,000
1925 . . . . .	4,910,000	2,000,000	6,910,000
1926 . . . . .	4,848,000	2,140,000	6,988,000
1927 . . . . .	4,265,000	1,895,000	6,160,000

Some 15 factories in Italy manufacture synthetic dyestuffs, and of these 10 produce sulphide dyes. The more important are in the province of Milan (eight), and in the provinces of Turin, Cuneo and Bergamo.

## Dyestuffs Markets: The Month's Business in Review

### From Our Own Correspondents

#### Lancashire

THE increased application of rayons or artificial silks to the production of fabrics is resulting in new problems for the dyer, the solutions of which are only likely to be effected by combined effort of weaver, dyestuff manufacturer, dyer and finisher.

The beauty of appearance and high textile qualities of many of the new crepe fabrics ensures for these fabrics an important and prominent place in textile manufacture, and consequently there is a powerful inducement to the dyer and finisher to overcome troubles which arise owing to the difficulty of penetration of hard-twisted crepe yarns and the overcoming of faults such as liability to crease, more especially in all-rayon cloths. For example, in the dyeing of satin face 100 per cent. acetate cloths, the only practical method at present is to dye these on slow-moving jigger dyeing machines in which the cloth is held out in open-width.

Under these conditions the dyestuffs of the S.R.A., or other dispersed type, do not very readily go on to the acetate silk, although in conjunction with cotton, or in contact with a cotton end-fent, the dyestuffs, being brought into more continuously intimate contact with the acetate, go on readily enough. It would appear that there is a need for water-soluble acetate-dyeing dyestuffs of good fastness to light and to washing.

The present fashion in printed artificial silk fabrics has given rise to problems which have been mentioned here before,

such as the shortage of suitable navys and blacks on acetate capable of giving white discharges. The method frequently adopted of first saponifying the acetate silk and afterwards printing by the method usual for cotton and viscose unions has obvious disadvantages.

The demand for dyestuffs in Lancashire has been appreciably better during the past month than during October, and there are indications of continuance of an increased demand.

During the past month cloth sales have been made in better quantity, and woven cloth is coming forward to the dyer and finisher.

There are no movements of importance in regard to prices.

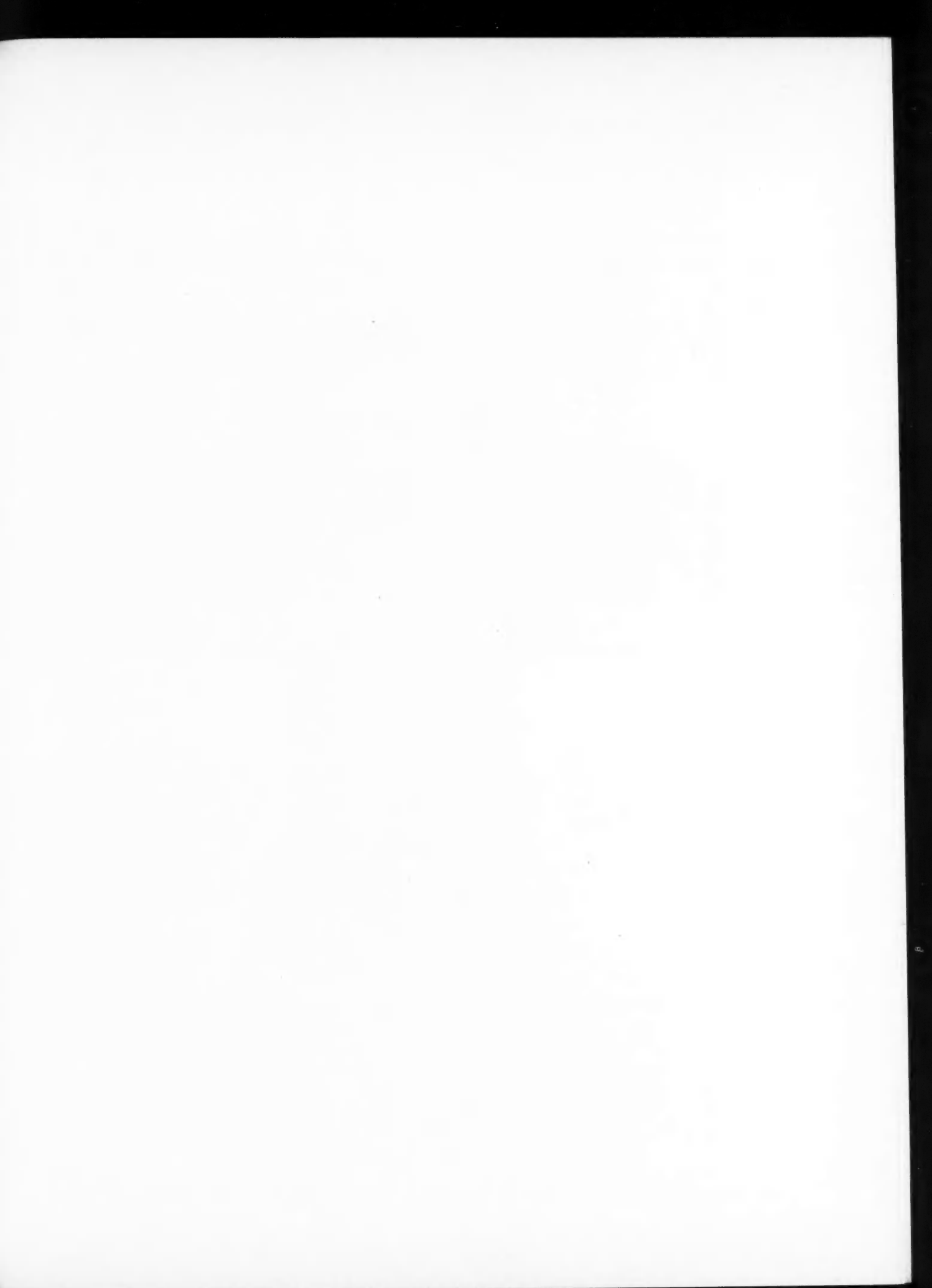
#### Scotland

Conditions in the textile industry in Scotland during November have shown little change. Wool prices having hardened, orders in the tweed and allied sections have shown some improvement, although so far this winter's orders are reported to be not up to the level reached at the corresponding time last year.

Round the Glasgow and Paisley districts conditions are somewhat patchy; some firms have sufficient orders to keep them going on full time and others only have a few days' work in sight.

The carpet trade is fairly satisfactory and calico printers are moderately well employed.

Dyestuff sales during the month are up to the average, with little change in price, and chemical markets have also been fairly uniform.

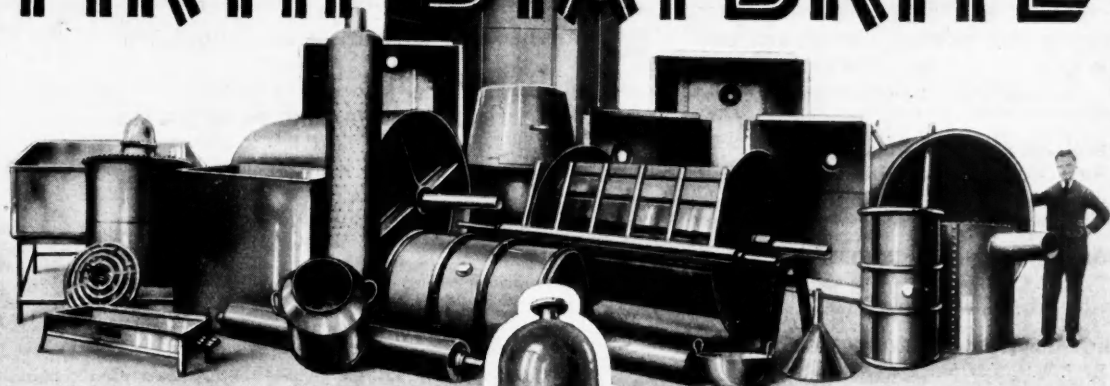




# Metallurgical Section

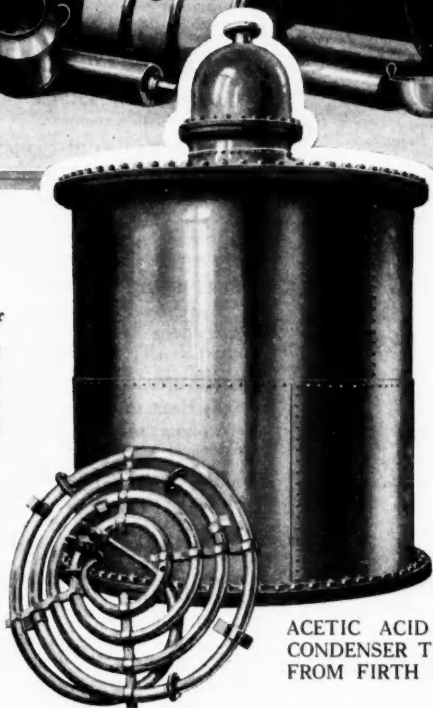
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# Monthly Metallurgical Section

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**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

## Some Alloys of Aluminium

By G. Malcolm Dyson, Ph.D., A.I.C.

Aluminium, which now occupies the fourth place in the production list of metals, is manufactured to the extent of 220,000 metric tons per annum, much of which is used, not as the metal itself, but in the form of various alloys, the use of which becomes necessary in order to overcome various defects which arise from the use of aluminium alone.

VARIOUS grades of aluminium are obtainable in the commercial market; the purest averages about 99.5 per cent. of aluminium, but the so-called "foundry ingot" is between 98 and 99 per cent. pure. The analysis below is averaged from a number of samples of each grade:—

	HIGHEST. Per cent.	FOUNDRY INGOT. Per cent.
Aluminium .....	99.50	98.01
Copper .....	0.048	0.04
Iron .....	0.207	0.88
Silicon .....	0.124	0.26
Nitrogen .....	0.044	—
Zinc .....	0.008	0.72

It will be seen that the "foundry ingot" contains about 1 per cent. of iron, an impurity which has an important effect on the grain size of castings. A third variety of aluminium met with in foundry practice is remelted scrap aluminium. This product has been the cause of much trouble and difficulty in the production of sound castings. In the first place, it frequently happens that some lead becomes mixed with the aluminium scrap and gets into the melting pot; lead does not alloy with aluminium, but remains in the melted metal as liquid globules, leading to soft spots in the subsequent castings. A second difficulty with remelted aluminium scrap is the fact that too high a temperature is often used for the melting, with the result that oxidation takes place, with consequent incorporation of oxide into the final melt, leading to small crystals of alumina in the casting. These lead to loss of strength and, by reason of their abrasive quality, to difficulties in mechanical working. In the working and melting of aluminium and its alloys a pyrometer should always be used, and in casting the lowest workable temperature should be maintained.

### "12" Alloy

Pure aluminium of the highest grade is but little used except for the production of aluminium vessels for the chemical industry, and for certain minor uses, such as the manufacture of cooking utensils. The most-used alloy is an aluminium-copper product known as "12" alloy. This alloy contains 92 per cent. of aluminium and 8 per cent. of copper, and is used for the production of vacuum cleaner parts, kettles, cameras, auto-body parts, containers, etc., where a strong and light alloy is required. Its specific gravity is 3.0, it melts at about 620° C., has a tensile strength of 21,000 lb./sq. in., a compression strength of 38,000 lb./sq. in., and a Brinell hardness of 60.

In the preparation of "12" alloy, the order of melting and adding the constituents is important; the copper should be melted under charcoal in a separate crucible and added to the aluminium at a temperature of 650° C. In stirring alloys of this type, there should be used an iron bar which has been coated with alundum cement; in this way contamination of the alloy with iron is avoided. Another way of making this alloy is to prepare a copper-rich aluminium parent alloy by melting copper under charcoal and adding bars of solid aluminium in weight equal to that of the copper used. These should be pushed down to the bottom of the crucible with the stirring bar and not allowed to float on the top, since this encourages oxidation. The copper-rich alloy can then be melted as required, and added to molten aluminium.

The temperature used for remelting "12" alloy previous to casting should not vary much between 650° and 700° C.; if allowed to rise to 800°, as may occur when no pyrometer is used, bad castings are obtained owing to contraction; in addition, oxide and nitride formation take place, and lead to inclusions and weakness, while blowholes are frequent from the carbon dioxide and monoxide absorbed at the higher temperature and released just prior to solidification.

There are many variants of "12" alloy. An alloy containing 85 per cent. of aluminium, 14 per cent. of copper, and 1 per cent. of manganese is used as a light alloy for pistons, but suffers from the fact that it has a large cooling contraction. Attempts have been made to avoid this by the use of an aluminium bronze, containing a small amount of magnesium, but without any outstanding success. A favourite alloy is that containing 87.5 per cent. of aluminium, 12 of copper and 0.5 of magnesium. This finds some use in the manufacture of light auto-engine parts.

### Nickel in Aluminium Bronzes

The addition of nickel to the aluminium bronzes just described leads to the formation of a better and harder alloy. Nickel is usually added in the form of "standard cupro-nickel," which contains 80 per cent. of copper and 20 of nickel. The total amount of nickel added is usually not more than 4 per cent. Among the aluminium bronzes there are three which stand out above the others—namely, Alpax, Lynite, and "Y" alloy. Alpax (which derives its name from that of its inventor, Aladar Pacs) is a silicon-aluminium bronze. There are various constitutions that have been suggested and used for this alloy, varying from 8 to 13 per cent. of silicon, and one of the most used is that containing 11 per cent. of silicon and 4 per cent. of copper. Alpax has an abnormal strength, which in the first melting is between 18,000 and 21,000 lb./sq. in., but this figure can be raised to 25,000 lb./sq. in. by melting under a flux containing sodium fluoride. The rise in strength appears to be due to the reduction in grain size which is brought about by the action of the flux.

The well-known alloy "Lynite" is a nickel alloy containing iron and other metals as shown below:—

	LYNITE. Per cent.	"Y" ALLOY. Per cent.
Aluminium .....	96	92.5
Copper .....	2	4
Nickel .....	0.2	2
Iron .....	1	—
Silicon .....	0.4	—
Magnesium .....	0.4	1.5

The preparation of Lynite is preceded by the preparation of a special "hardener." To prepare this, 10 parts of copper are added to 1½ parts of nickel, and the two melted together. Five parts of aluminium are added, and 3½ parts of tin-plate, the whole being stirred at this point until quite homogeneous; a further 10 parts of aluminium are then added. To prepare the alloy itself, 83½ lb. of aluminium is melted at a very dull red heat; 6 lb. of the hardener are added and ½ lb. of magnesium, followed by 10 lb. of 5 per cent. silicon-aluminium and a further ½ lb. of magnesium. The composition of an alloy prepared in this way is given above. Its ultimate strength is 27,000–29,000 lb./sq. in., the yield point 12,000–14,000 lb./sq. in., and the elongation in a 2 in. test

piece from 2.5-4 per cent. Such high mechanical qualities make Lynite very well suited for the manufacture of auto-engine pistons, and aluminium worms, where a high resistance to wear is necessary. "Y" alloy (its constitution is shown above) is remarkable for the fact that it is amenable to heat treatment and develops a tensile strength after such treatment of 20 tons/sq. in. In addition it will resist corrosion fairly well and for this reason has been used for aero parts. A short summary of some of the principal alloys of this class is given in Table I below:—

PISTON ALLOYS FOR CHILLED MOULDS.

Alloy.	Al.	Cu.	Zn.	Mg.	Mn.	Ni.	Fe.
Curtis .....	96	2.5	—	1.5	—	—	—
British .....	95	—	—	—	—	5	—
Mag. .....	95	2.5	—	1	—	1.5	—
U.S. Air Board .....	93	4	and	Si. 3%	—	—	—
N.P.L. .....	92.5	4	—	1.5	—	2	—
Rolls-Royce .....	92.5	5	1.2	—	—	0.5	Sb. 0.8
Lynite No. II .....	88.5	10	—	0.5	—	—	—
Brit. Air Board I .....	88	12	—	—	—	—	—
Aerolite .....	86	12	—	—	2	—	—
Brit. Air Board II .....	85	14	—	—	1	—	—
Benz .....	80	6	12	Trace	Trace	—	1.5

TABLE I.

## Duralumins

The term duralumin has almost become a generic name for a large series of aluminium-copper alloys containing magnesium. The so-called simple duralumin has the constitution copper 3.5-4.5 per cent., magnesium 0.5-1.0 per cent. and an

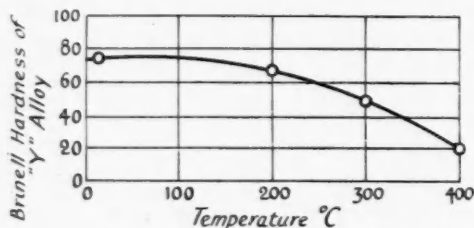


FIG. 1. ALTERATION OF BRINELL HARDNESS OF "Y" ALLOY ON HEATING AND COOLING.

equal quantity of manganese; the remainder is aluminium. This has been to a large extent replaced by "compound duralumin," which has a composition similar to that of the previously described alloy, save that the amount of copper is reduced to 2.5 per cent. and part of the aluminium (to the extent of 20 per cent. of the total alloy) is replaced by zinc. This alloy is also amenable to heat treatment; when quenched from 400° C. it shows a tensile strength up to 75,000 lb./sq. in. The outstanding property of the compound duralumin is its "age-hardening" qualities. On allowing it to stand it increases in hardness over a period of several days. It appears that on quenching a supersaturated solution of magnesium in aluminium is formed, and that this slowly crystallises, giving a tough, fine-grained structure. Subsequent heating, however, will remove the hardness, as may be seen from Figure 1. above, which gives the alteration in Brinell hardness of "Y" alloy on heating and cooling.

## Magnesium Alloys of Aluminium

Magnesium has a pronounced effect on the mechanical qualities of aluminium; it increases the fineness of grain, and scavenges out the oxygen and nitrogen present in the form of aluminium oxide and nitride. An 8 per cent. admixture of magnesium increases the tensile strength to 28,000 lb./sq. in. and the elongation on a 2 in. test piece to 5-10 per cent. Such an alloy has a density of 2.5, and can be easily prepared by adding the magnesium in sticks to aluminium which has just been melted. The sticks must be steadily depressed under the surface of the molten metal with tongs, or else there is risk of firing. The true compound between magnesium and aluminium has the chemical composition  $Al_3Mg_4$  and contains 54.5 per cent. of magnesium. It appears to be stable in moist air.

The effect of magnesium on "12" alloy is to reduce the contraction on solidification. On the other hand, the ternary copper-magnesium-aluminium alloys are more readily corroded, but have the advantage that they are more susceptible

to heat treatment. They are best prepared for use by quenching from 500° C. and ageing.

The true magnesium-aluminium alloys contain a preponderance of magnesium, and are derived as modifications of Elektron alloy, which is free from aluminium (see Table II).

MAGNESIUM-ALUMINIUM ALLOYS.

	Elektron.	Dow D.	Dow T.
Aluminium .....	—	8.5	2
Magnesium .....	95	88.5	92
Copper .....	0.5	2	4
Zinc .....	4.5	1	2

TABLE II.

Elektron alloy has a tensile strength of 18 tons/sq. in. and an elongation of 20 per cent. The yield point is 12 tons/sq. in. It is to be particularly noted that the specific gravity is only 1.8. The melting point is 630° C. and the alloy must not be melted in graphite pots, iron being recommended. The alloy machines wet. By the addition of aluminium to Elektron alloy, the Dow metals are obtained, as shown in Table II. They have approximately the same mechanical characteristics, but are slightly tougher than their prototype.

## Die-Casting Alloys

There are considerable difficulties in the way of the successful use of aluminium-containing alloys for the manufacture of die-castings of 2-3 lb. weight. In the first place, there is the wear and tear on the moulds, which seems to be intensified with aluminium; this has been overcome by the use of moulds of chromium-vanadium steel. Further, in the preparation of castings from zinc base alloys containing aluminium, oxidation and the presence of air lead to much wastage by blowhole formation; this difficulty has been overcome by the use of vacuum casting plant, in which the formation of blowholes is very much reduced. The composition of some of the die-casting alloys is given below:—

DIE-CASTING ALLOYS.

	Per Cent.		
Zinc .....	74	47	88
in .....	15	31	7
Copper .....	5	20	3.5
Aluminium .....	6	2	1.5

The last of the above alloys has the following characteristics:—M.P. 690° C., Brinell hardness 63, tensile strength 22,000/23,000 lb. sq.in., and specific gravity 7.16.

## Aluminium Solders

Ever since the first introduction of aluminium as a commercial metal, there has been difficulty in joining it in a satisfactory manner. Aluminium can be welded, but owing to the formation of an intractable film of oxide during the process, it is a difficult matter even for a skilled man to get a strong weld free from weakening oxide inclusions. Many formulae have been published, from time to time, for the preparation of aluminium solders, but they suffer from one (or both) of two serious defects; the join is either very weak mechanically, or else very readily attacked by moisture, with the result that it corrodes away in a very short time when exposed to the atmosphere. The following list of aluminium solders (quoted from Anderson on *Light Alloys*) is of considerable interest in showing the various types of solder:—

ALUMINIUM SOLDERS.

Name.	Composition.						Reduction	
	Sn.	Zn.	Pb.	Al.	Cu.	Others.	T.S.	Elong. in Area
Sterling .....	62	15	8	11	3	Sb. 1	13,000	1.6
Reisch .....	49	50	1	—	—	—	11,900	1.3
Crown .....	63	18	1	13	3	S. 2	14,300	1.9
Soluminium .....	55	33	—	11	1	—	9,800	1.5
Siefert .....	74	21	5	—	—	—	6,045	6.5
Richards .....	63	36	—	1	—	—	8,010	9.0
U.S. Bur. Stand. I .....	78	8	—	9	—	Cd. 5	14,300	18.0
U.S. Bur. Stand. II .....	86	9	—	5	—	—	12,000	8.0
U.S. Bur. Stand. III .....	—	75	—	5	—	Cd. 20	28,000	—
German .....	—	75	—	—	—	Cd. 25	—	—
Special .....	75	10	4.5	4.5	—	—	—	—

TABLE III.

As a final note, it may be added that an alloy of aluminium 45 per cent. and germanium 55 per cent. has been shown to give an ideal aluminium solder! The present price of germanium renders the use of such an alloy quite impossible in practice.

## Determination of Slag and Oxides in Wrought Iron

### From a Correspondent

ALTHOUGH probably not used to the same extent as it was formerly, wrought iron is still an important metallurgical product with many useful applications. One of its chief characteristics is its fibrous structure, which is largely due to the presence of mechanically entrapped slag. The very nature of the manufacturing processes renders this admixture of slag inevitable. The slag usually occurs in streaks elongated in the direction of working, and the micro-structures of wrought iron showing strings of slag inclusions in one direction and dots of slag in the structure of the material at right angles to this direction are familiar illustrations in text books of metallurgy. These slag inclusions and the form they take endow the material with very characteristic mechanical properties.

#### Importance of Determination of Slag

An ordinary chemical analysis does not yield information of much value, since it gives no indication of the distribution between the metal and slag of the elements determined, nor of the slag content. Since entrapped slag has so much bearing on the qualities of the material, the necessity of a method of estimating the extent to which it occurs is evident. It is an analytical problem which has exercised the minds of chemists for years, and various methods have been suggested from time to time. Apart from attempts made to separate slag and metal mechanically from crushed samples, the chemical methods have generally consisted of treating wrought iron borings with iodine, bromine, potassium bromide, potassium iodide, copper potassium chloride or mercury salts to dissolve away the iron and leave the slag as a residue; or of passing chlorine over the heated sample, when ferric chloride is formed and sublimed, leaving the slag behind.

Because of the volatilisation of some of the ferrous oxide, the latter method usually yields low results. Of the various forms of the first method, that of Eggertz, in which the sample is treated with iodine and the slag and oxide residue treated with hot water and dilute hydrochloric acid, was the most satisfactory.

#### An American Investigation

In the course of a thorough investigation of the question of the determination of slag and oxides in wrought iron, Wescott, Eckert and Einert (*Industrial and Engineering Chemistry*, 1927, **19**, 1285), examined many of the existing methods and developed a modification of the iodine method, which yields very satisfactory results. In the original iodine method of Eggertz, the mixture of the sample and cold water to which iodine is added is kept cold by surrounding the beaker with ice, and several hours are necessary to effect solution of the iron. It has been found, however, that it is not necessary to keep the solution cold or to add iodine gradually, and that the solution may be warmed and stirred continuously. In this way the time taken for solution of the sample is reduced from about seven hours to one hour. The objection concerning the time taken is therefore not a very vital one, and can be easily overcome, but another of a more serious nature affects the Eggertz method, namely, the washing of the residue with dilute hydrochloric acid. This reagent, it has been shown, has a marked solvent action on the slag, and therefore gives low results in estimating the slag content.

In the modified method, washing with hydrochloric acid is dispensed with, and the residue is washed with hot 10 per cent. potassium hydroxide solution and boiling distilled water. In the absence of standard samples of wrought iron, tests on the modified method and on the effect of various factors were made on prepared mixtures of plain low-carbon steel and puddle furnace tap cinder. Washing with potassium hydroxide is part of the procedure of both the original and modified methods, and as this in certain circumstances is liable to produce errors unless precautions are observed, special attention has been paid to it. It has been found that when the total phosphorus content is 0.150 per cent. or less, washing with caustic does not introduce an error of significant magnitude, but when the total phosphorus exceeds 0.150 per

cent., the use of caustic gives low results because of its solvent action on the slag phosphorus. If the caustic wash is omitted, the results are high if the total silicon exceeds 0.150 per cent., because of the oxidation of appreciable amounts of base metal or silicide silicon during solution in iodine. It is not possible to determine accurately the distribution of silicon between base metal and slag unless washing with alkali is adopted. When both phosphorus and silicon contents are high the amount of slag is calculated from determinations made with and without the alkali wash. Thus, in so far as washing the residues with alkali is concerned, the details of the procedure of estimating slag in wrought iron have to be varied in accordance with the phosphorus and silicon content of individual samples.

#### Experimental Details

With this provision the method given is as follows: To 3 grams of drillings in a 100 c.c. beaker 20 grams of resublimed iodine and 30 c.c. of distilled water are added. After the initial violent reaction the sides of the beaker are washed with a little distilled water, and the contents of the beaker are stirred vigorously by mechanical means for one to two hours. The mixture is then filtered by suction through a 9 or 11 cm. filter paper, and the filtrate washed with boiling distilled water until no trace of iodine is left on the paper and the washings give no iron reaction. The filtrate is then covered to the top of the funnel with boiling 10 per cent. potassium hydroxide solution, which is allowed to filter by gravity and not by suction. This alkali wash is carried out twice, after which the residue is washed with boiling water and filtered under suction, the washing being continued until no alkaline reaction to phenolphthalein is given by the washings. The filter paper and residue are dried, ignited, and weighed as slag and iron oxide.

A certain amount of caution is necessary in effecting the solution in iodine, and in the first stages it may be necessary to add cold distilled water to prevent its boiling over, but this is not liable to occur after the initial violent reaction. Silica is determined in the ignited residue of oxides and slag by dissolving in hydrochloric acid, evaporating to dryness and fuming with sulphuric acid. The salts are then taken up with water and nitric acid and the silica filtered off after boiling. After ignition, the residue is treated in the usual way with hydrofluoric acid. The percentage of silicon in the metal is given by the difference in the silica figure in the slag obtained in this way and the total silicon. Manganese in the slag is estimated in the filtrate from the silica determination after treatment with a slight excess of sodium bismuthate by titration with sodium arsenite.

M. C.

#### Canadian Aluminium Products Industry

THE Dominion Bureau of Statistics at Ottawa has forwarded to the High Commissioner of Canada in London a report indicating that the production of aluminium manufactures in Canada (except smelter products) was valued at \$3,192,696 in 1928. This output marked a new high level for the industry, being 38 per cent. above the total of \$2,318,894 for 1927. Fourteen plants, all in Ontario, manufactured aluminium articles in 1928. The capital employed by these concerns amounted to \$5,094,213, and the average number of employees was 609. Kitchenware of all kinds was made in 9 different factories, and 7 of these made kitchen utensils only. Two concerns made boot and shoe lasts; one made thermit and railway welding parts; and two firms made miscellaneous articles such as grocers' supplies and parts for automobiles, bicycles, washing machines, and skates. Imports of aluminium ingots, blocks, bars, etc., were valued at \$415,908 in 1928; tubing, \$46,430; leaf and foil, \$233,011, over half of which came from Germany; kitchen and household hollow-ware, \$284,740, mostly from the United States; and other manufactures, \$1,110,090, also largely from the United States. Exports in 1928 included aluminium scrap, \$249,066; bars, blocks, etc., \$8,049,367; kitchen utensils, \$46,384; and other manufactures, \$712,874.

## Metallurgical Topics: Monthly Notes and Comments

### From Our Own Correspondents

#### The Corrosion Research Report

Those interested in corrosion tests will read the Third (Experimental) Report to the Atmospheric Corrosion Research Committee of the British Non-Ferrous Metals Research Association, presented by Dr. Hudson, with extreme interest. It relates to field tests at stations where widely-differing atmospheric conditions prevailed, and brings out the marked influence of seasonal variations. The results obtained may be sharply distinguished from the conclusions which have been most carefully and discriminatingly drawn up by Dr. Hudson, as he has been cautious in their interpretation, and has himself drawn attention to sources of error. In the experiments the samples were placed in Stevenson screens (the louvered boxes in which meteorological instruments are often housed) and in such positions as inevitably to screen each other as well. Hence a positional correction is needed, although every effort was made to eliminate errors arising from this source, the samples being changed in relative position at intervals. Still, not only a screening, but a filtering effect must have prevailed, for it is reasonable to assume that in such conditions a corroding atmosphere could readily be stripped selectionally of its corroding constituents while passing successively over a series of coiled wires of different metals and alloys, such as were employed. Moreover, what is one metal's bane may be another metal's protection, and in such circumstances the oxide and other films, the formation of which may form protective coatings over the underlying metals (or in some cases act in just the opposite way, and accelerate corrosion), must inevitably differ according to their position and the time they have remained in it, and hence exert entirely specific and differential influences when their positions, later on, are changed or rotated. To deduce, from the results obtained, a "life" of the metal, seems, in these circumstances, of doubtful safety or value. In justice to Dr. Hudson, it should be said that he qualifies the conclusions extensively, and by the use of such phrases as "admittedly more or less hypothetical" and "necessarily approximate" shows very clearly that he claims no more, either for his results or conclusions, than is warranted by his own careful and painstaking observations.

#### Nitrogen-Hardening of Steel

THE effect of nitrogen in steel has been studied for many years past, and it has invariably been found that its influence is injurious, making the metal both red-short and brittle. On the other hand, the effect of cementing or case-hardening steel in an atmosphere of nitrogen, and thereby conferring upon it a very high degree of hardness, has only been studied comparatively recently, and only within the last year or so has it been applied with successful results. The nitrated steels of commerce are a valuable contribution to the machine shop, hence the article by Shun-Ichi-Satoh, a translation into French of which has recently appeared in the *Revue de Métallurgie* (Vol. 26, No. 5), will be welcomed as showing what occurs on "nitration," of which so much has been heard of late. Cementing has been tried by treating steel in ammonia gas; in sodium nitrate; in nitrogen itself; and in a number of other ways. The author's own experiments bore on electrolytic iron, an iron containing aluminium, titanium and manganese, and complex chromium steels containing from 0.612 to 1.938 per cent. of chromium, and, in addition, aluminium, titanium, manganese, zirconium, molybdenum, tungsten and vanadium, a medley of constituents which it might have been preferable to avoid. The samples were subjected in an electrical resistance furnace to the action of ammonia gas at 580° C. for 4½ hours. The Brinell hardness before and after treatment was determined.

#### Chromium and Titanium Nitrides

As to what happens, Shun-Ichi-Satoh states that the noxious influence of nitrogen in steel increases, according to the views generally held, in proportion to the carbon content. Nitrogen has the property of maintaining the  $\gamma$ -state of iron, and thus of retaining the carbon in solid solution and lowering the  $A_1$  point. In nitrating steel by means of sodium nitrate, it is difficult to get the nitrogen to penetrate a high-carbon steel

owing to the presence of a larger amount of solid solution of carbon than ordinarily occurs in  $\alpha$ -iron. When chromium is heated in nitrogen or in ammonia CrN is formed, and thus the total nitrogen percentage of a steel containing chromium increases. Similarly, a chromium steel heated *in vacuo* liberates more nitrogen than other steels. Hence chromium steels lend themselves with special facility to nitrational methods.

In the case of the chromium steel which was the subject of the author's own experiments, the Brinell hardness of the steel containing the lowest percentage of chromium was before nitrating 142, and after nitrating 248, or a difference of +106. In the case of the higher chromium steel (1.938 per cent. Cr.), the hardness rose from 229, prior to treatment, to 477, or the very considerable increase of +248. These differences pale in comparison with some of those attained in steels containing other special constituents. Thus, a steel containing 0.254 per cent. carbon, 2.246 per cent. Cr., and 1.00 per cent. Ti, had before treatment a Brinell hardness of 126. After treatment, the hardness number was 800, an increase of no less than 674. This is because, like chromium, titanium easily combines with nitrogen, and titanium nitride possesses exceptional hardness (910 Brinell). When, however, more than 3 per cent. of titanium is present, the case formed is exceptionally brittle, and liable to peel off. In the experiments described, the next highest results, so far as increased hardness is concerned, were obtained by nitrating aluminium steels containing chromium. Thus, a steel containing 0.217 per cent. carbon, 2.015 chromium and 1.927 aluminium, with an original Brinell number of 136, had its hardness increased by 589 to 725. Although the difference before and after treatment was smaller in the case of a steel containing 0.248 per cent. carbon, 1.710 chromium and 0.556 aluminium, the ultimate Brinell hardness after treatment reached no less a value than 815.

#### Magnesium

THE work on magnesium included in Bulletin No. 346 of the Bureau of Standards, Washington ("Light Metals and Alloys") is the best hitherto published on this metal. Meanwhile it continues to occupy space in foreign journals, and a useful contribution entitled "Magnesium and Its Alloys" has been appearing in that useful contemporary French publication, *Aciers Speciaux, Métaux et Alliages*. It is by R. Cazaud, and it summarises recent work done on magnesium and magnesium alloys in automobile and aeronautical construction. Hitherto the liability of these metals to corrosion has, it is said, impeded progress in their use: commercial varieties are prone to retain chlorides derived from the process of their extraction, and in an anhydrous form such chlorides possess a fatal affinity for moisture. Free from chlorides, on the other hand, the corrosion danger is much over-rated; pure magnesium assumes a protective film which is superior even to that of aluminium. Additions of calcium, cerium, etc., greatly improve both the mechanical properties of magnesium and its corrosion-resistance. Cadmium, in small amounts, likewise improves the soundness of magnesium ingots, and increases their malleability. With 5 per cent. of this metal added to the melt, a tensile strength of 21.3 kilogrammes per sq. mm. and an elongation of 10.2 per cent. can be obtained on annealed sheets of otherwise pure magnesium.

#### The Electroplaters' Society

THE concluding meeting of the Electroplaters' and Depositors' Technical Society's (E.P.D.T.S.) fourth session, held last month, inaugurated a new feature which other societies might with advantage copy. It had, too, other features of interest. To begin with, it marked the conclusion of Mr. Field's presidency, which he has held ever since the Society was founded. This cannot be other than a subject of profound regret, for Mr. Field has been an ideal president, and whatever the future of the Society may be, its success has hitherto been due in a very great measure to his handling and direction of its affairs. His knowledge of electroplating, both in theory and in practice, is extensive and sound, and his knowledge and services have been placed unreservedly at the service of the Society as a whole and of its members individually.

He has had, as he himself would be the first to admit, the ungrudging help of excellent officers and an excellent committee. The zeal of the first hon. secretary, Mr. Harrison, was ceaseless, and he has been worthily succeeded by the present energetic and devoted hon. secretary, Mr. Wernick. Still, it has been to its president that the Society has owed most, and we do not doubt that in the future, as in the past, Mr. Field will continue in his earnest work of promoting its interests and furthering its already high reputation, which he has done so much to establish and consolidate.

Dr. Hutton, the new president, has already done a great deal for the Society, while the new vice-president, Mr. D. J. MacNaughtan, has been second only to the late president in his indefatigable efforts for the Society, to which he has, from time to time, contributed very valuable papers, the lucidity of style of which, as well as his mastery of his subject, have already rendered him very popular with the members. The hon. treasurer, Mr. James, will continue to serve in that capacity.

### **A New Kind of "Open Discussion"**

To revert to the innovation referred to above, and introduced at the last meeting of the "E.P.D.T.S.," this took the form of a general discussion on subjects chosen by the members themselves, and assumed the form of questions and answers. The meetings of scientific bodies are as a rule convened to hear papers which have been selected by their councils, with only a vague reference to subjects on which the members themselves seek information, or would like to discuss. There are, of course, occasions when symposia are held by societies on some broad general subject, but the opportunity afforded to members of raising any subject they like, and informally discussing it amongst themselves is, we believe, quite novel, and differs entirely from the questions asked on a definite paper in the ensuing discussion, which, if not strictly relevant, would, of course, be cut of order. That the discussion in question was chiefly confined to matters relating to nickel plating in no way affects the principle involved, namely, that any kind of question might have been asked. An "open night" of this kind would, we feel sure, be popular with the members of most societies, who, as a rule, have far too few opportunities of meeting together to exchange views on subjects of immediate interest to them as distinct from some one specific subject which may appeal to a few only of those present.

### **Notes on Electroplating Plant**

SOME remarks made by Mr. J. W. Perring in a recent paper on "Electroplating Plant" were of great interest.

Mr. Perring said there are a number of materials that could be used for making vats or containing vessels for electroplating solutions; their use was determined by cost, ease of manufacture and suitability for the solutions they were to contain. They ranged in size from a few gallons to 50 feet or more long, as follows:—Wood vats without linings; slate vats; stone-ware and glass, round and rectangular; cast iron, enamelled and plain; iron, riveted and welded; lead-lined wood vats; asphalt or hard rubber; ebonite-lined vats; concrete or brick.

There were a number of methods of heating solutions: Steam, gas, thermosiphon and electricity. Solutions were usually required to be worked at about the following temperatures:—Cold 15° C., warm 100° F. (37·8° C.), hot 40–50° C. Cold solutions could be maintained at fairly constant temperatures by keeping the shop at an even temperature, especially at night; if more attention were paid to this, many of the troubles attributed to the weather might be eliminated.

Warm solutions could be heated by steam, gas, electricity or thermosiphon, hot water, coal, coke or gas being used to heat the boiler. For hot solutions, electricity was too costly, and steam or gas was usually used.

### **Steam, Gas and Electric Heating**

STEAM heating was probably the cheapest method of all. The boiler was complete with the necessary mountings and apparatus for filling (feed pump or injector), working at about 30 or 40 lb. per square inch, with the main steam pipe near the shortest and simplest route, to enable all the vats to be conveniently picked up. The main steam line should be arranged with a slight fall towards where the steam was required. If the line was "trapped," "water hammer" was likely to occur, with consequent liability of damage to pipes and fittings. Steam could be allowed to pass direct into the

solution, and the noise could be prevented by means of a special fitment. This method was convenient for hot water tanks, but not solution, as the steam condensed and gradually filled the vat.

As regards gas heating, gas could be burned under a suitable steam boiler, but would probably be dearer than coal. Iron tanks were usually heated by means of circular or straight rows of Bunsen flames, the top of the luminous cone being level with, or just below, the bottom of the tank. Lead-lined vats could be heated by means of a lead tube with a sealed bottom containing an inverted Bunsen burner. The tube was suspended vertically in the solution, actually a gas immersion heater. The thermosiphon principle could also be used.

Electric heaters were usually of the immersion type, the outer casing being made of a material not affected by the solution. This provided an ideal method of maintaining solution at constant temperature throughout the 24 hours, especially where only about blood temperature was required, e.g., building up with nickel. Immersion heaters with special casings, e.g., silica, etc., were fairly expensive.

In designing electric heaters one met with the following difficulties:—The actual element must be insulated electrically from the solution. Unfortunately, most suitably electric insulators were also good thermal insulators; thus it was necessary to force the heat through a poor conductor, which meant that the element could not dissipate its heat so quickly; an insulating material of high dielectric strength was chosen, the insulation could be made very thin. Mica answered this requirement, even when the element had to run much hotter than the solution. A number of nickel-cased immersion heaters gave trouble due to corrosion. These were placed in larger containers containing oil, and were still in use after some years.

### **Congress of Metallurgists at Dusseldorf**

THE forthcoming congress of metallurgists to be held at Düsseldorf, September 9–12, promises to be of unusual interest. Already nearly 200 persons from all over Europe, and some from America, have indicated their intention to participate in the gathering. The congress will comprise (a) the presentation and discussion of about 15 scientific and practical papers relating to metallurgical work; (b) visits to works in or near Düsseldorf and Berlin; (c) receptions, dances and other social functions. Those who desire to participate in the congress should write for particulars without delay to the Secretary of the Institute of Metals, Mr. G. Shaw Scott, M.Sc., 36, Victoria Street, London, S.W.1.

Members will assemble in Düsseldorf on Monday, September 9. In the afternoon, the eighth Annual Autumn Lecture will be delivered by Dr. A. G. C. Gwyer, on "Aluminium and its Alloys." On Tuesday, September 10, the morning will be occupied by the reading and discussion of papers, the afternoon being devoted to visits to works. In the evening the City of Düsseldorf will entertain members and their ladies to dinner. On the morning of Wednesday, September 11, further papers will be presented for discussion and the business meetings will be brought to a conclusion. The afternoon will be devoted to visits to works, and in the evening there will be a cabaret and dance in the Rheingoldsaal. For Thursday, September 12, an all-dry trip will be arranged to Essen to visit the works of Friedrich Krupp, Essen. The party will return to Düsseldorf in time to allow members who must return at once to England to travel by the night boat train. For other members extensions are being arranged, one including a trip through Holland and another a visit to Berlin.

### **Bureau of Information on Nickel**

IN order to increase the efficiency of its work, by avoiding overlapping and duplication of activities, the Bureau of Information on Nickel, Ltd., is joining forces with the Research and Development Department of the Mond Nickel Company, Ltd. It is believed that this rearrangement will provide greater possibilities of service on the part of each organisation, to British industry as a whole, and to all those who are interested in the use of nickel and its alloys. The title and address of the Bureau are now as follows: The Bureau of Information on Nickel of the Mond Nickel Company, Ltd., Imperial Chemical House, Millbank, London, S.W.1. (Telephone: Victoria 4444). The management of the Bureau will continue to be in the hands of Mr. A. C. Sturney.

## Trade, Commerce, Finance : The Month in Review

*From Our Market Correspondent*

ALTHOUGH a month has elapsed since the general election, the iron and steel trade has hardly settled down to its usual activity, and, as we are now at the beginning of the summer season, it is no use expecting any real improvement until the latter part of the year. In addition to this seasonal influence, there is no doubt that the change in the Government has been responsible for the quieter tone which has prevailed during the past few weeks. With a limited majority, the Government may be expected to walk warily, and it is reasonable to hope that there will be a minimum of interference with industry.

### Comparison with Former Years

The present condition of the iron and steel trade must be regarded as satisfactory in comparison with past years. There is a cheerful tone in the market and a fair amount of business is passing through to the mills. Some sections are exceptionally busy, such as small bars and semi-finished steel, while others are doing moderately well. Nevertheless, it is still essential that there should be a much bigger volume of trade to feed all the works. Production is still too much in excess of demand, particularly in plates. Most of the plate mills are working from hand to mouth, as is proved by the quickness with which delivery of new orders can be obtained.

The Steel Associations are giving serious consideration to this matter. It is clearly recognised that the available trade in this country falls far short of satisfying all the plate mills, and the only other source is the foreign markets. To get a larger hold of them will necessitate a drastic reduction of export prices. The export makers are prepared to face that if by so doing they can lift their outputs to a figure which will give them the economical working for which the mills were designed. For some time past the associated steel makers have had an arrangement which has enabled them to compete more readily in the overseas markets, and in the light of this experience they are putting into operation a scheme which should more effectively fulfil this purpose. If this is successful, and there is no reason why it should not be, it will benefit the steel trade all round.

### Activity in Pig Iron

A noticeable feature of the trade during the past month has been the activity in the pig iron market. In each district the reports show that the supply is not equal to the demand, and this is especially so in the Cleveland district. Makers have sold their outputs as far ahead as September and October, and the prices have steadily advanced. There has been little increase in production, as the additional furnaces which have been blown in are producing basic iron and hematite. Stocks of foundry iron are practically cleared out, and consumers in the Middlesbrough district have been obliged to purchase heavy tonnages from Northamptonshire and Derbyshire.

The difficulty in the way of increasing outputs in the Cleveland district is the shortage of ores and coke. Until a better supply of these is ensured the pig iron makers are moving cautiously and are in no hurry to increase the output. In the Midlands district, the Central Pig Iron Association, which has recently been formed to control the selling prices, is working satisfactorily, and has had the effect of bringing forward much new business. Consumers know that any movement in price will be upwards, and they are anxious to buy forward at the present price. It is quite likely that there will be a further advance before long, as the price of blast furnace coke is still hardening. The continued shortage puts the power into the hands of the coke makers, and they are insisting upon advances up to 1s. per ton for the renewal of contracts.

### Bright Spots

As we have already stated, the brightest sections of the steel trade are the billets and small bars. There is not a large quantity of Continental semi-finished steel being offered in this country, and the orders are mostly being placed with the British steel works. The billet mills generally have good order books, and consequently prices remain firm. It is a welcome change from the time, not so long ago, when billets were obtainable at absurdly low prices, owing to the foreign and internal competition. Small bars are still in strong demand,

particularly the qualities which are used in the motor trade. In ordinary qualities there has been rather more competition from abroad, and prices have eased slightly.

On the whole, however, steel prices remain unchanged. The Steel Associations, at their meeting in June, decided not to alter the official prices, which are £8 2s. 6d. for sections and £8 15s. to £8 17s. 6d. for plates, according to district. Boiler plates are still being sold at prices varying from £9 12s. 6d. to £10, and have not yet followed the advance in ordinary plates.

### A New Steel

A new steel which is coming well to the fore is the copper-bearing steel. This contains a small percentage of copper, usually up to 0.5 per cent., which renders the steel less liable to corrosion. It has been in use widely in America for some time, and is now finding favour with wagon builders and similar trades in this country. The railway companies are ordering it in fairly large quantities. The copper has no detrimental effect on the steel.

The output of pig iron in May was 654,800 tons, compared with 611,300 tons in April, there being seven more furnaces in blast. The output of steel was 840,400 tons, compared with 808,600 tons in April.

### Crucibles for High-Temperature Work

METALLURGISTS performing experimental work at high temperatures will probably be interested in the result of work done at the South-west Experiment Station of the United States Bureau of Mines, Department of Commerce, Tucson, Arizona, in developing crucibles for holding molten sulphides at very high temperatures.

In the course of a comprehensive investigation of the reduction of magnetite by molten sulphides, undertaken in co-operation with the University of Arizona, it became necessary to devise a method of making crucibles of a suitable material which would hold such melts, and the following technique was finally evolved. Pure electrically-fused magnesium oxide, mixed with 3 per cent. alumina, was ground to pass 350 mesh. A suitable quantity of the mixed oxides was moistened with 7 per cent., by weight, of 25 per cent. magnesium chloride solution, and tamped firmly into a 3-piece mould lined with heavy paper to form a slug having the shape of the outside of the crucible desired. Then the mould was placed in the chuck of a lathe and the slug was hollowed out to form a crucible which, after drying, was fired in a high-frequency induction furnace at a temperature increasing over a period of two hours from that of the room up to 2,100° C.

Crucibles so prepared are very dense, and will hold a molten mixture of iron sulphides and oxides for 45 minutes at a temperature of 1,500° C. without appreciable seepage. The melt will take up in that time about 2.5 per cent. magnesium oxide. This may be considered very satisfactory when one takes into account the temperature, the corrosive nature of the melt, and the relatively large surface presented to the melt by very small crucibles. Among other refractory materials whose properties were investigated were thoria, zirconia, chromite, and alumina. The three former were readily attacked by sulphides at high temperatures, and alumina did not resist the action of molten iron oxides well.

### The I.G. and Steel

JUDGING by developments which are at present taking place, it would seem that the I.G. Farbenindustrie A.-G. (the German Dye Trust) will soon be playing a very important part in steel production. The transaction by which the I.G. has acquired 40 per cent. of the capital of the Rheinmetall A.-G. is now complete. The latter company is the only concern permitted under the Treaty of Versailles to produce certain limited quantities of materials for the Navy, and is also a producer of fine steel railway materials, etc. The I.G. has provided one-third of the capital for the new iron and steel works at Bochum, which will be completed in the autumn. It is also making efforts to obtain control of a small steel sheet works in Westphalia. The I.G. is now making most of the steel tubes for its chemical works.

## Some Inventions of the Month

### By Our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature, published weekly in THE CHEMICAL AGE.

#### Alloys

AN alloy consisting of a tungsten or molybdenum carbide and an element of lower melting point, such as nickel, chromium, cobalt, iron, or silicon, is obtained by melting an alloy of the refractory metal with the other element and carbon, pulverising the product, and sintering it at 2,200–2,400° C. See Patent Application 310,885, bearing the International Convention date May 2, 1928, by F. Krupp, Akt.-Ges., of Essen, Germany.

ALUMINIUM alloys, suitable for making normalisable castings, contain copper, together with titanium, zirconium, or boron, and in some cases small amounts of magnesium, zinc, silicon, manganese, chromium, or iron. The castings may be subjected to an after-treatment, comprising heating for some hours at 510–520° C., cooling, and ageing either naturally or at 100–150° C. See Patent Application 309,586, bearing the International Convention date April 13, 1928, by O. Reuleaux, of Frankfurt-on-Main, Germany.

#### Cadmium Plating

ACCORDING to Patent Applications 309,071–2, bearing the International Convention date April 4, 1928, by C. H. Humphries, of Kokomo, Indiana, U.S.A., (1) barley sugar, caramel, malt sugar, or malt syrup, is added as a brightening agent to an acid cadmium plating bath; (2) a cadmium anode and a lead or like insoluble cathode are used in an acid bath, and their areas are so proportioned that the cadmium content of the bath remains constant.

#### Copper

ARTICLES made of copper or copper alloys are rendered resistant to corrosion by embedding them in a powder comprising one or more of the elements chromium, tungsten, molybdenum, tantalum, manganese, silicon, nickel, cobalt, vanadium, titanium, uranium, or zirconium, and heating for 5–15 hours in a neutral or reducing atmosphere at 500–1,000° C. Alternatively the articles may be coated electrolytically and similarly heated. See Patent Application 308,353, bearing the International Convention date March 22, 1928, by J. Laissus, of Paris.

IN a process for refining copper described in Specification 310,356, dated January 16, 1928, by H. H. Alexander, of Westfield, New Jersey, U.S.A., oxide of copper contained in a mass of the molten metal is reduced by injecting under pressure a solid carbonaceous reducing agent, *e.g.*, coal, charcoal, or coke, in finely divided form. The reducing agent is preferably introduced at several points of the molten mass by means of steam or a gas under pressure. The process is particularly applicable in place of the usual "poling" process, for the reduction of copper which has been oxidised together with the impurities in a previous refining operation.

#### Extracting Metals

A PROCESS for extracting metals, particularly iron, from their ores, described in Patent Application 309,998, bearing the International Convention date April 19, 1928, by A. Langer, of Vienna, comprises the continuous reduction of the ores in presence of a stream of the molten metal. A mixture of the ore and a reducing agent, *e.g.*, coal, introduced at one end of an open-hearth furnace, travels in opposite direction to the molten metal introduced at the other end, and the metal finally passes under a slag baffle to an outlet. Part of this metal may be returned to the opposite end of the furnace to constitute the stream of metal.

CHROMIUM, manganese, and vanadium, low in carbon and silicon, are obtained by exothermic reaction of an oxygen compound of the metal with a low-carbon silicon alloy or silicide of the metal. The slag produced may be treated, *e.g.*, by smelting in an electric furnace with carbon and silica, with or without addition of metal, to obtain a silicon alloy suitable for use in the exothermic reaction. Specified oxygen compounds are chromic anhydride, chromates, manganese dioxide, vanadium pentoxide and vanadates. See Patent Application 309,594, bearing the International Convention date April 13, 1928, by Electro Metallurgical Co., of New York.

#### Iron

IN a pneumatic process for dephosphorising iron, described in Patent Application 310,468, bearing the International

Convention date April 26, 1928, by P. Ries and F. Bicheroux, of Liège, Belgium, the dephosphorising is effected simultaneously with decarbonisation and without any after-blow by injecting pulverised lime during the decarburisation. The lime is preferably carried by air or inert gas supplied to the converter through tuyères arranged below the ordinary blast belt.

A PROCESS for case-hardening molybdenum-iron or -steel by nitrogenisation is described in Specification 311,588, dated July 4, 1928, by J. L. F. Vogel, of Widnes, Lancashire. The metal is heated with one or more solid compounds of nitrogen which (at or below the temperature of nitrogenisation of iron by nascent nitrogen) decompose, in presence of the metal with or without contact with air, water, or water vapour with production of nascent nitrogen. Specified nitrogen compounds are urea, cyanides, cyanamides, sodamide, potassamide, hexamethylenetetramine, naphthylenediamine, dicyandiamide, phenylhydrazine, acetamide, and guanidine. The treatment, which is usually of several hours' duration, is preferably effected at a temperature below 1,100° C., to avoid any deformation of the metal.

CAST iron of superior machinability is made from molten iron by the use of metallic calcium as a graphitising catalyst, according to Specification 312,126 (Elkington), dated February 20, 1928, a communication from Meehanite Metal Corporation, of Chattanooga, Tennessee, U.S.A. To molten iron containing more than 2 per cent. of combined carbon, or which would contain that amount of combined carbon when cast, there is added a quantity of metallic calcium beyond that which may be neutralised by acidic or other constituents, thus leaving a surplus of the calcium sufficient for the degree of graphitisation desired. An average of about 8 oz. of calcium to 100 lb. of metal is required. In addition to the calcium, magnesium, barium, strontium, or lithium may be added.

#### Magnesium

THE resistance to corrosion of magnesium and its alloys is increased by treatment for one hour or more with a boiling aqueous solution of a bichromate. See Patent Application 305,197, bearing the International Convention date February 2, 1928, by I.G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany.

To protect a jet of molten magnesium, or of a molten alloy with a high magnesium content, from further oxidation, particularly in casting operations, the film forming on the surface of the metal is preserved in a coherent form. Thus in pouring the metal from a ladle into a pool to form a casting, the mouth of the ladle is maintained in a fixed position relatively to the gate of the mould to avoid any breaking strain on the surface film. The formation of a coherent film may be assisted by supplying to the atmosphere surrounding the molten metal substances, *e.g.*, a spray of finely divided sulphur, adapted to combine therewith. See Specification 311,998 (Mond), dated August 9, 1928, a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany.

#### Tin

A PROCESS for the extraction of tin, described in Specification 310,639, dated April 14, 1928, by H. L. Sulman and H. F. K. Picard, of London, is of the type in which the tin is recovered as oxide fume obtained by subjecting a heated mixture of the ores, concentrates, etc., with carbonaceous material to a current of air. The process is specially applicable to the treatment of low-grade concentrates containing substantial amounts of iron, arsenic, and other metals or minerals, which would be deleterious when smelting in the ordinary manner, and is characterised by the fact that the charge in an open porous condition is so disposed (*e.g.*, in a layer of suitable thickness on a grate, air being passed through) that oxidising conditions are maintained throughout the charge. The composition of the charge and the temperature conditions are maintained such that any production of molten slag is avoided, and an admixture of pyrites or like sulphur-bearing material is found to assist the recovery of the tin as oxide. In some cases it is advantageous to sulphidise a portion or the whole of the tin mineral present by a preliminary heat treatment. The construction and operation of the furnace plant and of the devices for collecting the tin oxide from the fume are described and illustrated in detail.

## Current Articles Worth Noting

*We give below a brief index to current articles in the technical Press dealing with metallurgical subjects.*

**ALLOYS.**—The critical dispersion of Lantal. H. Bohner. *Zeitschrift Metallkunde*, May, pp. 160-165 (in German). Noteworthy connections have been found between tensile strength and electrical conductivity in relation to the temperature and time of hardening. The technical significance of the results is discussed.

Dilatometric investigations of thermal effects in the annealing of duralumin and its structural components. M. Haas and H. Hecker. *Zeitschrift Metallkunde*, May, pp. 166-173 (in German). The following structural systems were investigated: Al-Cu, Al-Mg<sub>2</sub>Si, Al-Cu-Mg<sub>2</sub>Si, as well as the German duralumins 681, 681B, 681B<sub>4</sub>.

**ALUMINIUM.**—The röntgenographic investigation of aluminium at high temperatures. A. J. Alichanow. *Zeitschrift Metallkunde*, April, p. 127 (in German). An X-ray investigation to determine whether aluminium undergoes an allotropic change between 575° and 600° C. showed that the structure remains unaltered (face-centred cube).

**ANALYSIS.**—The analysis of speculum metal. L. Schweitzer. *Chemiker-Zeitung*, June 12, pp. 457-459 (in German).

The determination of sulphur in unalloyed steels, alloy steels, pig iron, and iron alloys by combustion in a stream of oxygen. K. Swoboda. *Zeitschrift analytische Chem.*, Vol. 77, Parts 7-8, pp. 269-277 (in German).

The determination of vanadium in steel. I. Kassler. *Zeitschrift analytische Chem.*, Vol. 77, Parts 7-8, pp. 290-298 (in German). The ether separation is avoided by treating the steel with dilute sulphuric acid, the small part of the vanadium which dissolves being precipitated by shaking with zinc oxide suspended in water. The determination may be completed in three hours.

**BRASS.**—The effect of third metals on the constitution of brass alloys. I. The influence of lead. O. Bauer and M. Hansen. *Zeitschrift Metallkunde*, May, pp. 145-151; June, pp. 190-196 (in German). The ternary system Pb-Zn-Cu has been investigated in the region of 100 to 25 per cent. of copper and up to 2.5 per cent. of lead, by thermal and microscopic means.

The effect of aluminium, lead, iron and tin in brass. Report of the committee on brass of the German Metallurgy Society. *Zeitschrift Metallkunde*, May, pp. 152-159 (in German). Eleven series of alloys were investigated for the effect of the above metals on various mechanical properties.

**CHROMIUM PLATING.**—Eliminating health hazards in chromium plating. J. J. Bloomfield. *Chem. & Met. Eng.*, June, p. 351.

**COPPER.**—Crystalline changes in copper due to annealing. F. C. Howard and F. T. Dunn. *Ind. Eng. Chem.*, June, pp. 550-553. Crystalline changes upon annealing a rod of cold-worked copper have been investigated, using a strong etching reagent. The laminated structure of etched crystals of copper, which because of its regularity is probably due to some physical property, has been photographed at relatively high magnification.

**CORROSION.**—The prevention of corrosion of tubes. F. Besig. *Korrosion und Metallschutz*, May, pp. 99-110 (in German).

Investigations on the corrosion of the metals of power vehicles through fuels or fuel mixtures. K. R. Dietrich. *Korrosion und Metallschutz*, May, pp. 110-114 (in German).

The action of protective coatings on aluminium alloys. H. Röhrig. *Korrosion und Metallschutz*, April, pp. 85-88 (in German).

**CRYSTAL STRUCTURE.**—X-ray pattern of metallic crystals. G. B. Deodhar. *Nature*, June 15, p. 909.

**GENERAL.**—Magnetic alloys of iron, nickel, and cobalt. G. W. Elmen. *Journal Franklin Inst.*, May, pp. 583-617.

Graphitisation in the presence of nickel: A contribution to the study of graphitisation. H. A. Schwartz. *Trans. Amer. Soc. Steel Treating*, June, pp. 957-970. The quantitative relations of time, temperature, nickel

concentration and graphite formed are discussed, and silicon is shown to be inoperative as an accelerator in the presence of nickel.

Torsional modulus of carbon steel, phosphor bronze, brass and monel metal. W. P. Wood. *Trans. Amer. Soc. Steel Treating*, June, pp. 971-985. Results of numerous modulus determinations are given. It is concluded that the factors appearing in the usual spring deflection formula have little or no influence on the determination of the modulus.

**MAGNESIUM.**—High purity magnesium produced by sublimation. H. E. Bakken. *Chem. and Met. Eng.*, June, pp. 345-347.

**PASSIVITY.**—The theory of passivity phenomena. V. The effect of covering layers on the potential of a metal. W. J. Müller. *Monatshefte*, Vol. 52, Part 1, pp. 53-58 (in German).

**STEEL, ALLOY.**—A study on the constitution of high manganese steels. V. N. Krivobok. *Trans. Amer. Soc. Steel Treating*, June, pp. 893-956. Discusses the constituents of austenitic manganese steel (Hadfield steel) under different conditions of treatment. The cold working of this steel, followed by heating to certain temperatures, causes decomposition of the original austenite.

The application of science to the steel industry. IV.—W. H. Hatfield. *Trans. Amer. Soc. Steel Treating*, June, pp. 986-1,026. Deals with special steels (wear-resisting, magnet, and non-magnetic steels, steels of low coefficient of expansion, steels of high electrical resistance, and causes of failure); corrosion—and acid-resisting steels (corrosion tests, mechanical and other properties as affecting application, chromium-nickel steels).

### Development of Chilean Iron Industry

In order to free local industries from the need for foreign supplies of raw materials, the Chilean Government has enacted a law which authorises the President to invest, for the account of the State, 40,000,000 Chilean pesos (about £6,000,000) in taking up two-thirds of the shares in a corporation which is being formed in Southern Chile for the production of iron and steel. The company, which will have five Government directors out of seven, proposes to build electric blast furnaces near Port Carral, and hopes within 18 months to be able to deliver iron and steel rails, bars, plates, and wire, thereby cutting down substantially the importation of iron and steel products from the United States and Europe, which now amounts to nearly 230,000 tons yearly.

## Commercial Intelligence

*The following are taken from printed reports, but we cannot be responsible for any errors that may occur.*

### Mortgages and Charges

[NOTE.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debts due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case, the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.]

**CREIGHTON (T. R.), LTD.**, London, E., ironfounders. Registered May 30, £1,700 and £2,288 10s. Land Registry charges, to T. R. Creighton, The Foundry, Stepney Causeway, and Mrs. C. F. Creighton, Lucerne, The Ridgeway, Chingford; charged on properties at Limehouse, etc.

**GALLOIS LEAD AND ZINC MINES, LTD.**, London, E.—Registered May 31, £700 debentures, part of £20,000; general charge (excluding plant, etc., where the purchase price is not fully paid).

**WIGPOOL COAL AND IRON CO., LTD.**, Gloucester.—Registered May 29, £3,000 A debentures dated December 14, 1927, to September 3, 1928, and £2,000 and £500 B debentures dated January 5 and April 3, 1929, parts of £5,000 and £5,000; general charge. \*£26,000. April 4, 1929.





## Monthly Metallurgical Section

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**NOTICE.**—Communications relating to editorial matter for our *Monthly Metallurgical Section* should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

### *The International Relationship of Minerals* Sir Thomas Holland's Remarkable Address to the British Association

*An unusual address, culminating in a suggestion that the British Empire and the United States were in a position (through their control of minerals) to enforce peace if they acted together, was delivered on Wednesday at Johannesburg to the British Association for the Advancement of Science, by Sir Thomas Holland, the President. The more important parts of the address, which was entitled "The International Relationship of Minerals," are given below.*

A FEW years ago members of this Association looked forward annually to a generalised statement of the results of their President's own research work in science. The rapid specialisation of science, with its consequent terminology, has, however, made it increasingly more difficult in recent years for any worker to express himself to his fellow members.

Coincident with the excessive degree of specialisation which has developed with embarrassing rapidity within the present century, the problems of the war drew scientific workers from their laboratories and forced them to face problems of applied science of wider human interest. And the atmosphere of this great mining field (the Witwatersrand) stirs ideas of this wider sort—ideas concerning a field of human activity which, in recent years, has affected the course of civilised evolution more profoundly than seems to be recognised even by students of mineral economics. This must be my excuse for inviting you to consider the special ways in which the trend of mineral exploitation since the war has placed a new meaning on our international relationships.

#### **Minerals and Peace**

With knowledge of the shortcomings which were felt during the war, in variety as well as quantity of metals, it was natural immediately after to review our resources, with the object in view of obtaining security for the future. But events have since developed rapidly, both in international relationships and in mineral technology. The evolution of metallurgy during the present century, and the developments in mining on which metallurgy depend, have placed new and rigid limitations on a nation's ability to undertake and maintain a war; consequently, the control of the mineral industries may be made an insurance for peace. Let us first consider briefly how these circumstances have arisen, how each country has passed from the stage of being self-contained in variety of essential products to the most recent of all developments, the change to large-scale production that has tended to the concentration of the mineral and metal industries to certain specially favoured regions which will hold the position of dominance for several generations to come.

Metals, we know, have been used since early times for simple implements and weapons, but it was not until the industrial revolution in Great Britain that the mechanisation of industries led to any considerable development of our mineral resources, first slowly and with a limited range of products, then on a large scale and with an extended variety.

#### **The Steel Industry**

It is obvious that the growth, if not with equal certainty the origin, of the industrial revolution, was due to the close association of suitable minerals in England. It was because non-phosphoric ores were still available that, at a later stage, Bessemer was able to give that new impetus which increased the lead of the English steel maker; and so, when Thomas and Gilchrist came still later, with their invention of a basic process applicable to pig-iron made from phosphoric ores, their invention fell on barren soil in Britain.

The new process, however, found applications elsewhere, and, instead of adding to the stability of the English steel industry, it gave the United States the very tonic they required, whilst the industrialists of Germany—where political stability had by then been established—found the opportunity

of developing the enormous phosphoric ore deposits of Alsace-Lorraine, which had been borrowed from France eight years before. And so it was through the genius of Sidney Gilchrist Thomas, and his cousin, Percy Carlyle Gilchrist, that Germany was enabled in 1914 to try the fortune of war.

For the first half century after the industrial revolution, Great Britain was able to raise its own relatively small requirements of iron as well as of the other metals that consequently came into wider use—copper, zinc, lead and tin. The rapid expansion in steel production which followed Bessemer's announcement of his invention at the Cheltenham meeting of the British Association in 1856, brought with it the necessity of going further afield for the accessory ores and for further supplies of non-phosphoric iron ores.

#### **Alloys**

The next important step in metallurgical advance came in 1888, when Sir Robert Hadfield produced his special manganese-steel; for this led to the production of other ferro-alloys, and so extended our requirements in commercial quantities of metals which were previously of interest mainly in the laboratory—vanadium, tungsten, molybdenum, aluminium, chromium, cobalt and nickel. The adoption of alloys, especially the ferro-alloys, at the end of the last century, opened up a new period in the newly established mineral era of the world's history; for, besides the increase in the quantity of the commoner base metals which were wanted for the growing industries of Great Britain, it was necessary now to look further afield for supplies of those metals that had hitherto been regarded as rare in quantity and nominal in value.

The country in which the industrial revolution originated and gathered momentum, because of the close association of a few base metals, could no longer live on its own raw materials, and never again will do so. Even in peace time Great Britain alone consumes twice as much copper and just as much lead as the whole Empire produces. Meanwhile, developments had occurred elsewhere, notably in Germany, where political stability had been secured, and in the United States, where the Thomas-Gilchrist process also had stimulated expansion. Thus, by the beginning of the twentieth century, the industrial activities of the world had entered a new phase, which was characterised, if not yet dominated, by the necessity for minerals to maintain the expanding arts of peace.

#### **No Nation Self-Contained**

From this time on no nation could be self-contained; a new era of international dependence was inaugurated, but the extent and the significance of the change was not consciously realised by our public leaders until 1914, when it was found that the developments of peace had fundamentally changed the requirements for war. Indeed, not even the German General Staff, with all its methodical thoroughness, had formed what the tacticians call a true "appreciation of the situation."

Two illustrations of short-sightedness on both sides are sufficient for the present argument. Up to the outbreak of war, although the wolfram deposits of South Burma were worked almost entirely by British companies, the whole of the mineral went to Germany for the manufacture of the metal, tungsten, which was an essential constituent of high-speed tool steel. Sheffield still occupied a leading place in the

production of this variety of steel, but was dependent on Germany for the metal, which the Germans obtained mainly from British ore. Under the compulsion of necessity, and without consideration of commercial cost, we succeeded before the middle of 1915 in making tungsten, whilst Germany, failing to obtain an early and favourable decision in war, used up her stocks of imported ore and turned to the Norwegian molybdenum for a substitute, until this move again was partly countered by our purchase of the Norwegian output.

Germany then found that she wanted ten times more nickel than Central Europe could produce; so she imported her supplies from the Scandinavian countries, and they being neutral, obtained nickel from another neutral country, where the Canadian ores—the world's main source—had hitherto chiefly been smelted and refined. We thus realised, not only our dependence on other lands for the essential raw minerals, but we had the mortification of finding that, through our own previous shortcomings in the metallurgical industries, we were compelled to face lethal munitions made of metal obtained from our own ores.

### Mineral Distribution a Controlling Feature

The political boundaries of the nations, originally delimited on considerations dominantly agricultural in origin, have now no natural relation to the distribution of their minerals, which are nevertheless essential for the maintenance of industries in peace time as well as for the requirements of defence. This circumstance, as I hope to show in the sequel, gives a special meaning to measures recently designed on supplementary lines in Europe and America for the maintenance of international peace, measures which, as I also hope to show, can succeed only if the facts of mineral distribution become recognised as a controlling feature in future international dealings.

Since the industrial revolution in Great Britain, the increase of mechanisation and consequent consumption of metals has been accelerated with each decade. For example, in the year 1870 the United States produced 69,000 tons of steel; in 1880, 1½ million tons; in 1890, 4½ millions; in 1900, 10 millions and in 1928, 45 millions.

For the present we can assume with fair confidence that, taking the world as a whole, the depletion of natural stores is not yet alarming, although the rate of acceleration, by reason of its local variation, forces into prominence some international problems, which will influence, and, if effectively tackled, will facilitate, the efforts to stabilise conditions of international relations.

### Relative Abundance of the Metals

I have elsewhere made estimates of the quantities of metals stored in that part of the outer film of the earth's crust which may be regarded as reasonably accessible to the miner. The first feature of surprising interest to the man in the street is perhaps the relative abundance of those metals with which he is familiar in the arts—copper, lead, tin, zinc and nickel. There are, indeed, among the so-called rare metals some which are distinctly more abundant than lead, although this is the cheapest of the lot in price, and is consumed at the rate of over a million tons a year.

And so one gets at once an indication of two important features. Firstly, the miner works only those deposits in which the metal is concentrated sufficiently to make their exploitation a profitable business; and, secondly, the metalliferous ores vary greatly in the completeness with which they have been concentrated in special places to form workable ore-deposits. Nickel-ore, for instance, occurs under conditions which conspicuously hinder its freedom of local concentration. Indeed, nickel stands apart from the other metals, for, whilst it is important in peace time and is dangerously important during war, yet, under the present state of mining and metallurgical practice, the deposits in the world worth working for nickel can be numbered on the fingers of one hand, and nine-tenths of our supplies come from a single district in Canada.

### Exploitation of Low-Grade Ores

Our reference to nickel shows that the metalliferous ores vary in their degrees of concentration, and, therefore, in their suitability for working; but, as the result of estimates made for a few common metals, we shall not be far from the average in assuming that we shall never recover more than about one-millionth of the total that lies within workable distance

from the surface of our accessible dry land. And another conclusion, based on a similar group of calculations, shows that our greatest total tonnages are not contained in the rich deposits, but in those of low-grade.

It follows, therefore, that every advance in metallurgical science and in mining technology that makes it possible to work our low-grade ores adds appreciably to the actuarial value of civilisation; for our mineral resources can be worked once and once only in the history of the world, and when our supplies of metalliferous ores approach exhaustion, civilisation such as we have now developed during the last century must come to an end.

When a miner raises a supply of ore in concentrated form for the metallurgist, he damages, and so places beyond reach for ever, far larger quantities of residual ore than he makes available for use. When a metallurgist takes over the product of the miner and separates the refined metal for use in the arts, he also incurs serious losses, although not to the same extent. There are thus before both the miner and the metallurgist opportunities for extending the actuarial value of civilisation; and because the cost of labour is the principal constituent in the total bill, and has recently swamped contemporaneous advances in technology, the gradual elimination of manual labour by mechanisation is obviously the most profitable line of research.

But mechanisation carries with it in general a tendency to limit operations to the larger deposits, with the concurrent neglect of those propositions which are widely scattered over the earth, and, though individually small, represent in the aggregate a serious section of our limited resources. And our operations in mining, with the family of industries dependent on minerals, tend more and more to be restricted to a few special regions, where work can be done on a large scale. So now, with this thumb-nail sketch of the way in which the new mineral era is developing, we are free to examine more closely the influence which this change in the configuration of the industrial world is likely to have on international relationships.

### The British Empire and the United States

In the first place, it becomes obvious that no single country, not even the United States, is self-contained, whether for the requirements of peace or for the necessities of war. Not even the more scattered sections of the earth that are politically united to form the British Empire contain the full variety of those minerals that are the essential raw materials of our established activities. Between them, these two—the British Empire and the United States—produce over two-thirds of the 2,000 million tons of mineral that the world now consumes annually. Each of them has more than it wants of some minerals; but, in order to obtain its own requirements at economic rates, each finds it necessary to sell its surplus output to other nations. Each produces less than it wants of some minerals, and so must obtain supplies from other nations to keep its industries alive. Each of them is practically devoid of a few but not always the same minerals, which, though relatively small in quantity, are none the less essential links in the chain of industrial operations.

Even if these two could "pool" their resources they would still be compelled to obtain from other nations the residual few. For it is important to remember that, unlike organic substance, it is not possible to make synthetic metals, and it never will be; it is not even possible to make artificial substitutes for many essential minerals that are used as such and not merely for their metallic constituents. There is no other mineral and no artificial substance, for instance, that can combine the qualities which give to the mineral mica its position of importance in the arts. There will never be a synthetic mica.

### The New Civilisation

Thus the international exchange of minerals is an inevitable consequence of our new civilisation; and the cry for freedom of movement, for the "open door" and for equal opportunity for development comes into conflict with the unqualified formula of "self-determination." Whatever may have been possible before the industrial revolution, when the mineral industry merely contributed to the simple wants of agriculture, when most national units were self-contained, the formula of "self-determination" has come too late in the world's history to do good without a more than consequent amount of harm.

We cannot even live now without the free interchange of our minerals for those of other nations; in the name of civilisation we dare not go to war.

Primitive workers in various lands have opened up to relatively shallow depths rich but small deposits of ores, and in Eastern countries especially, where forms of civilisation extend far back into history, the numerous and widespread "old workings" have given rise to travellers' impressions of great mineral wealth. But low-grade deposits that the ancient miner could not utilise are now opened up by mechanical methods on a large scale; and, on the other hand, what satisfied the primitive metallurgist in abundance would be of little use to the modern furnace.

#### **Travellers' Tales**

We have now to revalue the tales of travellers which have had a dangerous influence on those who have directed the course of international competition.

For this reason it is necessary to review afresh the resources of the undeveloped Far East, which has for many years been regarded as a menace to Western industrial dominance. The vague general notion that mineral deposits are evenly distributed throughout the earth's crust has fed the impression that the development of China, which is much larger than the United States, may yet shift the centre of industrial gravity when her great population becomes awakened and organised by western technical science.

It is true that the people of the East are rapidly adopting the methods and using the mechanical facilities of western nations—railways, telegraphs, power factories, steel ships and other metal-consuming devices; but the critical investigations made by mining geologists, especially since the war, tend with a striking degree of unanimity towards recognising the remarkable circumstance that China, as well as other countries of the Far East, is deficient in those essential deposits of minerals on which our mechanised form of civilisation is based.

Exploratory work by mining geologists tends more and more to show that the essential mineral products are far from evenly distributed over the land areas of the world. Western Europe and North America have an undue share of those deposits that can be worked on a large scale, and it is the large-scale movement that marks the specialised character of the new industrialism. Anglo-Saxon character would have found limited scope for its energy but for the fact that nine-tenths of the coal, two-thirds of the copper and as much as 98 per cent. of the iron-ore consumed by the world come from the countries that border the North Atlantic.

#### **Political Control**

The industrial revolution, which began in Great Britain, has always been recognised as a dominant phase in western civilisation, but it is now assuming a new character. It spread first to the western countries of Europe, and developed there because of the favourable conditions of mineral resources, but the force of the movement faded out towards the Slavic East and the Latin South; the mechanical industries of Italy are based on imported scrap. When the new industries became transplanted west of the Atlantic the natural conditions which originally favoured Great Britain were found to be reproduced on a larger scale.

Thus, in these two main areas, separated by the Atlantic Ocean, a family of industries based on mineral resources has arisen to dominate the world; for no similar area, so far as our geological information tends to show, seems to combine the essential features in any other part of the world. Political control, which follows industrial dominance, must lie with the countries that border the North Atlantic.

#### **National Exclusiveness**

It is only in this region that there is any approach to the state of being self-contained. And yet since the war there has arisen, first in Europe and then by imitation in Asia, a degree of national exclusiveness more pronounced than any which marked international relations before 1914. Each small political unit has become vaguely conscious of the value of minerals, and has shown a tendency to conserve its resources for national exploitation on the assumption that they add appreciably to military security.

There is, however, no such thing now as equality of nations in mineral resources; "self-determination" and the "closed door" are misleading guides to the smaller nations. Political

control may hamper, but cannot stem, the current of the new industrialisation; commercial and industrial integrations are stretching across political boundary-lines; and the demand for the interchange of mineral products will be satisfied in spite of fiscal barriers.

#### **Science and Politics**

It would have been a shock to our members if, before the war, political problems were discussed from this chair, and party politics may always be inconsistent with the mental products of culture. But the results of science and technology now limit the effects of national ambitions, and therefore dominate the international political atmosphere for good or evil. One is justified always in suggesting non-controversial measures that tend to good; and this it is proposed to do very briefly as the direct suggestion of the new configuration of the mining and metallurgical world.

The League of Nations has accomplished a large measure of international understanding in questions of social value; its influence in forestalling possible causes of war has raised new hopes; but fortunately, so far, it has not been compelled to use any such instrument of force as a blockade, and any such measure that clashed with the vital economic considerations of first-class powers would probably cause stresses well beyond its elastic limits. The more recent and simpler pact of Paris associated with the name of Mr. F. B. Kellogg wants equally an ultimate instrument for its practical enforcement.

#### **Prevention of War**

It was with this ultimate object in mind that the outline of my argument was drafted after the Glasgow meeting last year; but I am glad to find that my views have since been expressed independently. Senator Capper, of Kansas, in February last, submitted a resolution to the American Legislature recognising this shortcoming of the simple treaty, and proposing to supplement its moral obligations by a corollary which, if passed, will empower the Government on behalf of the United States to refuse munitions to any nation that breaks the multilateral treaty for the renunciation of war.

Senator Capper's resolution, however, still leaves unsolved a residual problem of practical importance. Those of us who had the painful duty of deciding between civil and military necessities in the Great War, know well that there is now but little real difference between the materials required to maintain an army on a war footing and those that are essential to the necessary activities of the civilian population; materials essential for one purpose can be converted to articles required for the other.

Thus, if Senator Capper's resolution be adopted by those who have signed the Kellogg Treaty, either sympathy for the civil population would be stirred, or the armies would be still supplied with many essential munitions. The definition of "conditional contraband" would still remain as a cause for international friction. It is suggested, therefore, as an amendment to Senator Capper's resolution, that the simple words "mineral products" be substituted for "arms, munitions, implements of war or other articles for use in war."

#### **Enforcement of the Kellogg Pact**

The only two nations that can fight for long on their own natural resources are the British Empire and the United States. If they agree in refusing to export mineral products to those countries that infringe the Kellogg Pact, no war can last very long.

The confederation of American States has the advantage of forming a compact geographical unit, without inter-State fiscal barriers to hamper the interchange of mineral products. The British Empire, in the words of Principal Nicholas Murray Butler, "has passed by natural and splendid evolution into the British Commonwealth of Nations"; it is composed of geographically scattered and independent political units among which freedom of interchange, with due regard to local interests, can be effected safely only by more complete knowledge of our resources. Next year the Empire Congress of Mining and Metallurgy will meet in this city to discuss the proposition which I submitted to it at Montreal in 1927; and this address must be regarded, therefore, as an introduction to a movement which one hopes will supply the necessary data, and so facilitate a working agreement between the two great Mineral Powers that alone have the avowed desire and the ability to ensure the peace of the world.

## Metallurgical Topics: Monthly Notes and Comments

### From Our Own Correspondents

#### Duralumin in Aeronautical Engineering

DURALUMIN was much in evidence at Olympia during the International Aeronautical Exhibition and tended somewhat to overshadow the other metals, heavy and light, that enter into the structure of modern airplanes and seaplanes, as well as into that of the engines themselves. Duralumin has this in its favour, that with strength it combines lightness and a reasonable resistance to corrosion, three essential properties it is not easy to attain simultaneously in any other metal. The corrosion resistance is, however, considerably affected by the nature of the treatment to which the material has been subjected. A valuable article on the subject of duralumin generally has appeared in a recent number of the French journal, *Aciers Spéciaux, Métaux et Alliages*. The author, R. Cazaud, points out that the tensile strength of duralumin is fairly well maintained at reasonable temperatures, falling off by 10 per cent. at 100° C. and by 20 per cent. at 150°. At 300° C., however, the loss of strength is 60 per cent. It corrodes swiftly at such temperatures. Cazaud repeats the opinion that, from the point of view of corrosion by sea water, duralumin does not behave as well as pure aluminium, but the behaviour of varieties containing manganese is superior to duralumins which do not contain that constituent. Zinc accentuates corrosion, and Cazaud considers that copper has the same effect.

#### Blast Furnace Practice

THE second volume of Mr. Fred Clements's book, *Blast Furnace Practice*, Vol. II, *Design of Plant and Equipment* (Ernest Benn, Ltd., pp. 509, £3 3s.) has followed with commendable celerity the publication of the first volume, and we understand that Vol. III (*Operations: Utilisation of Subsidiary Products*) is well on the way. The two most important chapters in the new volume are probably those relating to skip and bucket charging (which has long been a subject of controversy between blast furnace men) and to the distribution of solid materials, concerning which there can hardly be two opinions. In respect of both matters, Mr. Clements's experience, and the pains he has been at to reap the experience of others for the benefit of his readers, entitles him to speak with authority, and to be heard with respect.

In regard to skip *versus* bucket-charging systems, it is plain that, correct distribution being the common end of all proper methods of blast furnace charging, and being attainable with either system, subsidiary conditions will determine which system to adopt. Mr. Clements has, in his time, held a brief for skip systems; on the Continent bucket systems are popular. There are and can be no hard and fast lines, and in any case Mr. Clements holds, in his book, an even balance, and treats the subject from the broadest possible point of view. The best of each system is described; the pros and cons are fairly stated, and, what is more helpful than any academic discussion as to relative merits, the subsidiary, yet really determining, considerations are given due weight.

#### Uniformity in Materials and Distribution

HALF the problems of charging, of irregular distribution of the stock, of uneven working, channelling, scaffolding, and the many troubles to which a blast furnace is prone, would be removed if it were economically feasible to grade the ore, coke and fluxes so that charges suitably sized, and of regular physical nature and chemical composition, could be uniformly fed to a furnace. Mr. Clements is an advocate of sizing, and in his earlier volume he has shown the advantages that accrue from this operation. In the volume now under discussion, he again emphasises the advantages of uniformity of materials, and when "rationalisation" spreads to blast furnace practice it is possible that groups of furnaces in any given vicinity, and working on similar ores for the production of similar grades of pig iron, may be able to pool their resources in bunker space, handling equipment, etc., and achieve a greater degree of grading to size, physical condition and chemical composition in their materials than heretofore. When that time comes a far higher efficiency and a far lower coke consumption may be anticipated. In the meantime, a blast

furnace manager can only use what he gets, as and when he gets it, and Mr. Clements's admirable text-book will be of help to him in making the best of things. It will do more: by placing before him the latest types of design and equipment, and all the data the author has collected, it will enable the technical staff to compare their practice with that of others and to ascertain just where there is room for improvement.

#### The British Association Meeting

THERE is a marked paucity of papers possessing a direct metallurgical interest among those presented at this year's meeting of the British Association in South Africa. The Association's most useful work, perhaps, is in helping to popularise science. It is one of the few bodies the proceedings of which attract interest in the daily Press, and the inference is that metallurgy, being essentially an expert art and science, is not as yet popular with the public, which is a pity. Sir Thomas Holland it is true, touched on the mineral wealth of South Africa in his interesting presidential address, but in view of that wealth, the exploitation of which must depend on metallurgy, it is to be regretted that metallurgy could not have been accorded a section to itself. It has hitherto, in meetings of the Association, been subsidiary to the Engineering Section, but this time there was not a single paper on a metallurgical subject in that section. With the exception of a practical paper by Mr. H. A. White, on "The Chemistry of Gold Extraction," in the Chemistry Section, only indirect reference to metals occurred, in papers read in the Mathematical and Physical Sciences Section. In view of the ignorance of the general public of the principles which underlie great basic industries, and the fact that Africa, besides producing gold and platinum, has untold wealth of copper, tin, chromium, manganese and iron, to say nothing of radium, a useful occasion of bringing home to the man in the street what our South African and West African possessions really count for in any scheme of Empire development would seem to have been missed.

#### Government Committee on Iron and Steel Industries

THE Government Committee to investigate the condition of the iron and steel industries will consist of Lord Sankey; Mr. Tom Shaw; Sir Cecil Budd; Mr. C. T. Cramp; and Sir William Plender. The terms of reference are:—"To consider and report on the present condition and prospects of the iron and steel industries, and to make recommendations as to any action which may appear desirable and practicable in order to improve the position of these industries in the markets of the world." The committee, as in the case of the inquiry into the cotton industry, will be a sub-committee of the Committee of Civil Research.

Lord Sankey, the Lord Chancellor, was chairman of the Coal Industry Commission in 1919. Mr. Tom Shaw is Minister of War. Sir Cecil Budd is chairman of the London Metal Exchange and managing director of the British Metal Corporation. He served on several committees of the Ministry of Munitions and the Board of Trade. Mr. C. T. Cramp is the general secretary to the National Union of Railwaymen. Sir William Plender is senior partner of the firm of Deloitte, Plender, Griffiths and Co., chartered accountants. He was president of the Institute of Chartered Accountants from 1910 to 1912.

#### American Fellowship for British Investigator

DR. E. B. SANIGAR, at present investigator in electro-metallurgy at Sheffield University, has been awarded by the American Electro-Chemical Society the Edward Weston Fellowship in Electro-Chemistry. Dr. Sanigar will be the first holder of this fellowship, and has elected to hold it at Columbia University, New York City. The award is made without distinction of sex, citizenship, race or residence. Dr. Sanigar is a graduate of Sheffield University. He was the first holder of a scholarship awarded by the Worshipful Company of Cutlers of London, and spent two years at the Charles University, Prague, Czecho-Slovakia, gaining the degree of

Doctor of Natural Science with the highest honours in 1926. Dr. Sanigar has been engaged on research work on silver plating, and has published several papers on this subject in scientific and technical journals.

### **Copper and Lead Precipitation by Sponge Iron**

It is easy enough to get sponge iron—operations having that object in view have inspired countless efforts in the past, all more or less attended with success. It is, moreover, the basis of the latest development in iron metallurgy. What to do with it when it has been obtained is the question. Hitherto it has not been a satisfactory source of iron *qua* iron. Its uses in other connections are, however, numerous, and the precipitation of lead and copper from solution by means of sponge iron is the subject of a very valuable report by the U.S. Department of Commerce (*Bulletin* 281). The sponge is used as a substitute for scrap, and as it exposes a far greater surface to the action of the liquors, it is more efficient. The bulletin embodies a research on the rates of diffusion of the metal ions in the solutions and through the metallic particles surrounding the sponge iron particles. In the case of lead precipitation, vigorously-stirred solutions heated to 60° C. are employed, when the metal is precipitated in about an hour. Porous and fresh varieties of sponge iron react faster than when the sponge is dense and impure. In the case of copper, for the precipitation of which iron has been used from time immemorial, sponge iron reacts with great speed and efficiency.

### **Industrial Furnace Technique**

THE publishing house of Ernest Benn has just issued *Industrial Furnace Technique*, translated from the Swedish of A. Hermansen (pp. 293, 25s.). Mr. Hermansen, well known as a designer and manufacturer of industrial furnaces, gives in this book the results of his special study and experience.

The chapter headings are as follows:—Temperature; heat quantity and specific heat; production of high temperatures; fuel; carbonisation; producer gas; transmission of heat; the dependence of time of heat transmission; the movement of gases; furnace building in general; regenerators and recuperators; burners and draught openings; working chambers; calculation of laboratory working space; materials for furnace building; supporting the furnace; starting up and tending the furnace; and heat balance.

### **Iron and Steel Manufacturers' Federation**

THE National Federation of Iron and Steel Manufacturers (Corporate Organisation), Ltd., was registered on Friday, July 26, as a company limited by guarantee, without share capital, with 500 members, each liable for one guinea in the event of winding-up. The objects are to promote the welfare of the iron and steel industry in the United Kingdom, and to co-operate in any manner that may be thought proper with, and generally to assist the operations of the voluntary organisation known as the National Federation of Iron and Steel Manufacturers; to certify the origin, material, mode of manufacture, quality, accuracy, and other characteristics of iron and steel and goods manufactured therefrom; to apply for, register, and own any trade marks to be used in connection with iron and steel, and iron and steel goods certified by the Organisation, etc.. The management is vested in an executive committee. The directors are: A. O. Peech; H. E. Parkes; E. J. Fox; B. Walmsley; J. Craif; J. Henderson; and H. C. Bond. The solicitor is R. B. Hopkins, 20, Park Row, Leeds.

### **Investigating the Applications of Tin**

THE formation of a Tin Applications Committee, to organise research into the improved commercial application of tin and the discovery of new uses for the metal, is announced. A considerable sum, it is stated, has been guaranteed by firms representing the tin mining and smelting industries to cover the outlay on a programme of research for two years ahead. The research work has been placed under the supervision of the British Non-ferrous Metals Research Association. The chief objects of the Tin Applications Committee are to collect and disseminate a greater knowledge of the properties of metallic tin, to investigate the scope of its industrial applications, to seek possible new uses, to keep manufacturers and consumers informed as to the results of research, and to encourage and support study and research.

### **Reduction of Cuprous Oxide by Carbon Monoxide**

THE bright annealing of copper is an industrial practice of considerable importance. In connection with this process, the equilibrium concentrations of carbon monoxide and dioxide which may exist in contact with copper and cuprous oxide at elevated temperatures, are of interest. Data obtained at the Pacific Experiment Station of the United States Bureau of Mines, Department of Commerce, Berkeley, California, in co-operation with the University of California, enables estimates of these equilibrium concentrations to be made in two independent ways.

Although the publication of final data on one method cannot take place until certain further experimental work is accomplished, repeated requests which the Bureau of Mines has had for data relative to the annealing and deoxidation of copper have brought about the issuance of a preliminary report, Serial 2926, "The Reduction of Cuprous Oxide by Carbon Monoxide," by C. G. Maier. This paper deals with the calculations of gas concentrations in the reduction of cuprous oxide, using more scientific data which will be published later, and refers especially to those conditions of temperature and concentration of practical implication, and from which critical limits ensue.

### **Technical Limits of Bright Annealing**

THE limits which must obtain technically in the bright annealing of copper when carbon monoxide is the reducer are defined in this paper. The possibility of the use of a carbon dioxide atmosphere containing small amounts of carbon monoxide for the deoxidation of copper both in the solid and liquid state is also suggested. If the oxide content of ordinary grade copper wire is eliminated, it is well known that an increase of 2 to 5 per cent. in electrical conductivity may be obtained. Present wire drawing practice often involves the use of steam for protection during annealing, and it is not impossible that substitution of carbon dioxide containing small amounts of carbon monoxide might result in considerable saving on the basis of pounds of copper per unit of conductivity, because it would permit some decrease in the original oxide content of the metal to be obtained.

Copies of Serial 2926 may be obtained from the United States Bureau of Mines, Department of Commerce, Washington, D.C.

### **The Sources of Tantalum**

IN a recent report on tantalum, the United States Bureau of Mines, Department of Commerce, says that its sources, uses and markets are very limited. Should an unforeseen and unexpectedly large supply of ore become available, it is not likely that the demand would increase in proportion; in fact, the price would probably fall unless the supply were closely controlled.

Tantalum is found in comparatively few minerals and under narrow geologic conditions. It is one of a pair of metals that are almost unknown separately, although columbium, the other member of the pair, is sometimes reported alone in minerals. The metals are very difficult to separate, possibly more so, excepting zirconium and hafnium, than any other pair of closely associated metals, such as gold and silver, rubidium and cesium, uranium and thorium, and the rare earth metals. The tantalum minerals have been found exclusively in pegmatites or in deposits closely related to the pegmatites, and outside the real pegmatites the deposits amount to little.

Although there is a fairly long list of tantalum minerals, few are of importance. Columbite is by far the most common and the most important; it is a very variable tantalate and columbate of iron and manganese, which may contain almost no tantalum or almost no columbium. If the quantity of columbium is very small, the mineral is known as tantalite. The content of iron and manganese may also vary. If a tantalite contains almost no manganese, it is dense black and is called tapiolite (also known as skogbolite); if manganese is present to the almost total exclusion of iron, the mineral is a beautiful translucent red and is called manganotantalite. Antimony or calcium (lime) may be present almost to the exclusion of manganese and iron, and the minerals are then known as stibiotantalite and calciotantalite respectively. The elements listed above are only the principal elements occurring in these minerals. All the tantalum-columbium minerals contain small quantities of other elements.

## **Trade, Commerce, Finance: The Month in Review**

**From Our Market Correspondent**

WE have now arrived at the season of the year when the reports of the condition of the iron and steel trade usually seem rather flat and uninteresting. The experience of long years past cannot be belied; and everyone who is actively connected with the industry knows that from the middle of July until early September trade is at its quietest.

### **Holidays and Repairs**

In the first place, the holiday spirit is abroad, and this has a very real influence on the volume of business done. Advantage is usually taken of the summer months to close down the works for a week or ten days and thus enable the employees to get a holiday. During the stoppage the opportunity is taken of overhauling the plant and machinery, which, after twelve months' more or less continuous operation, is showing signs of wear and tear, and it is reconditioned so as to cope with the demands of the coming year.

About this time each day's post brings notices from all parts of the country, from steel makers and consumers alike, of the closing down for holidays and repairs, and the number of these notices shows how widespread is the custom, as it must of necessity be. Consequently, in stating that business during the past few weeks has been quieter, and is likely to be more so in the month of August, one is not necessarily implying that the iron and steel trade in itself is actually worse. Indeed, it is gratifying to be able to point out that the falling-off this year is on the whole less marked than usual, and this may augur well for the end-of-the-year revival which seems to be generally expected.

### **The Pig Iron Market**

The activity in the pig iron market continues, notwithstanding the quieter tone in the other sections, although even in pig iron there is evidence of the holiday influence. The amount of business done and the inquiries sent out show that the consuming trades are still busy. In the Cleveland district the position is practically the same as reported last month. There is still a scarcity of supplies of foundry iron, and the iron masters are unable to augment the supply owing to the continued shortage of coke.

As might be expected, the scarcity of coke is reflected in the ever-increasing prices which the coke-makers demand. Each new purchase is at a higher price than the last. The weekly deliveries contracted for are rarely given, and the blast furnace owners are obliged to contract for considerably more tonnage than they actually require, in order to ensure adequate supplies for the furnaces, and even then it is not always easy to keep going at full output. In such circumstances it is not surprising that the price of pig iron remains firm, with a tendency to advance. Indeed, in the Midlands, the Central Pig Iron Producers' Association has just put up prices by 2s. 6d. per ton. That in itself is a good indication of the strength of the market, as it is not usual for prices to advance in the summer months. Pig iron from the Midlands is still being sent into the Cleveland district in considerable quantities.

The hematite trade is equally good, with a continued active demand for both home and abroad. It is confidently stated that prices will move upwards as soon as the works get into full swing again after the holidays. In the meantime, efforts are being made to increase the output by blowing in additional furnaces as soon as supplies of raw materials are available.

### **Steel**

The trade in semi-finished steel is comparatively good, although there is a decline in the amount of business done, partly owing to the holiday conditions and partly to the reduced demand. The Continental works are not in such a strong position as they were, and they are making renewed attempts to capture the English market, so far with little success. For most purposes there is still a strong preference for British steel. Even where buyers are inclined to take the Continental billets they are in no hurry to buy, as they are hopeful of further reductions which will make a purchase

more attractive. The home price for ordinary soft billets is about £6 15s. per ton, while the foreign price is under £6 per ton delivered.

The finished steel trade is quiet. Orders are coming in slowly and the output figures for the month will no doubt show a reduction, while for August still lower returns can be expected. Nevertheless, there is here and there evidence of a desire on the part of consumers to cover well ahead at present prices, as it is recognised that the present dullness is likely to be of short duration, and it is tempting to take advantage of the slight weakness which is manifest in certain sections of the industry. Small bars, for instance, are not quite so good a market as they were. The foreign prices have come down, and the re-rollers are finding it difficult to secure orders in competition. Even for the better qualities of steel which are bought from the British makers rather attractive prices are being quoted by some of the makers whose order books are lean.

### **Prices**

There is no change in the official prices of controlled materials, plates and sections. One hears of some very low prices being quoted for boiler plates, generally from the works which are short of specifications for the plate mills. It is unfortunate that there is no hope of any material increase in the demand for ship and tank plates, as there can be no competition among the makers for these owing to the price control, and consequently the better qualities, such as boiler plates, deep stamping steel, etc., are made the subject of very keen competition until the price is forced down almost to the level of the commonest steel.

The suspension by the Government of the work on the new warships will somewhat aggravate the position, as good contracts had been placed by the Admiralty with the various steel firms, and no further specifications against these will be forthcoming so long as the suspension is on.

### **The Coal Position**

An interesting position has arisen in regard to the collieries. The miners are pressing their demand for the shorter working hours, and the Government are holding them off as long as possible. The Prime Minister apparently does not want to make a move in this matter, as he knows quite well that the collieries cannot afford to bear any further increase in production costs, and until a way can be found to reimburse the coal owners for the cost of the shorter working day, the Government policy is to mark time.

The official returns for the coal mines show that in some districts there is a net loss on the working, whilst in those districts where a profit is made the amount is insignificant. Meanwhile the colliery owners are carrying on as usual, and they appear to be quite willing to book forward contracts at the prices which have prevailed during the first half of the year.

### **The Attitude of the Government**

The Government's intentions towards the iron and steel trade may be good, but so far they are vague. Safeguarding, as we know, is definitely shelved, and is not likely to be revived during the lifetime of the present régime. Instead, a committee has been appointed to inquire into the present position and prospects of the iron and steel industries, and to make recommendations as to any action which may appear desirable and practicable in order to improve the position of these industries in the markets of the world. It is strange that another committee should be appointed, when there is already available the Balfour Report, which deals very fully with the iron and steel industry. No attempt has yet been made to work out the recommendations of that report.

The production figures for June show an increase of six in the number of blast furnaces at work. The output of pig iron was 657,800 tons, compared with 654,800 tons in May. The output of steel was 830,900 tons, compared with 843,800 tons in May.

## Some Inventions of the Month By Our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature, published weekly in THE CHEMICAL AGE.

### Alloys

ALLOYS offering resistance to corrosion by chemical agents and to oxidation or scaling at high temperatures are described in Specification 313,471, dated March 10, 1928, by Sir R. A. Hadfield, of London. They have as principal ingredients: iron, carbon, chromium, nickel, tungsten, and copper, the combined chromium and nickel content being 18 to 32 per cent., that of the nickel being 2 to 20 per cent., and that of the chromium 5 to 30 per cent. Appreciable amounts of silicon and manganese may also be present.

### Detinning Scrap

TIN is recovered from tin-plate scrap by means of a solution of hydrochloric acid or of a chloride such as stannic or ferric chloride, with continuous or intermittent regeneration of the solution by the addition of an oxyacid of chlorine, such as chloric or perchloric acid, or salts thereof. The resulting solutions contain 5 to 15 per cent. of tin, which may be recovered by electrolysis. The scrap may be pre-treated by cleaning with caustic potash followed by immersion in hydrochloric acid. See Patent Application 312,924, bearing the International Convention date June 2, 1928, by L. Ughetti-La Corsa, of Turin, Italy.

### Iron

A METHOD of hardening iron or steel is described in Specification 314,219, dated June 26, 1928, by R. Esnault-Pelterie, of London. The metal is first subjected to a mechanical hardening process, and thereafter to the action of gaseous ammonia or of a compound which liberates gaseous ammonia or nitrogen on the application of heat. A preliminary tempering operation may be employed to ensure uniform hardness of the metal prior to the mechanical hardening. The treatment with gaseous ammonia or the like is preferably effected in a muffle or other closed chamber heated to 200 to 700° C., and the temperature, pressure, or duration of the treatment may be varied to regulate the degree of hardness imparted. The process is specially suitable for hardening balls, rollers, and races for bearings.

NON-CORROSIVE and heat-resisting surfaces are obtained upon iron by treatment at a temperature above 800° C. (preferably about 1,000° C.) with an aluminium-chromium alloy not mixed with other solid chemically inert material, such treatment being effected in the absence of oxygen or of gases, such as carbon monoxide or carbon dioxide, having an oxidising action under the conditions of working. An atmosphere of hydrogen or of a suitable gas mixture containing it, e.g., a mixture of nitrogen and hydrogen, is preferred. In an example, the use of an alloy containing 85 per cent. of chromium and 15 per cent. of aluminium at 1,000° C. for four to fourteen days in an atmosphere of pure hydrogen is specified. See Specification 314,314 (Johnson), dated March 19, 1928, a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany.

A PROCESS for removing phosphorus and sulphur from pig iron without oxidising the carbon is described in Patent Application 312,361, bearing the International Convention date May 25, 1928, by F. Krupp Akt.-Ges., of Essen, Germany. Carbon and a dephosphorising and desulphurising slag are added successively or in admixture to the molten metal contained in an open hearth or rotary tubular furnace or in the forehearth or hearth of a shaft furnace. The slag contains fining means such as iron or manganese oxides, as well as oxides of alkaline or heavy metals required to fix the oxides of phosphorus.

### Nickel

A PROCESS for the recovery of nickel by electro-deposition from nickel sulphate solutions obtained in the treatment of ores, concentrates, residues, etc., is described in Specification 314,579, dated March 30, 1928, by S. C. Smith, of London. Solutions of low acidity are employed with insoluble anodes, and undue increase of acidity, which would inhibit further deposition of nickel, is prevented by the continuous or intermittent addition of ammonia to the bath during the electrolysis, thus converting the sulphuric acid into ammonium

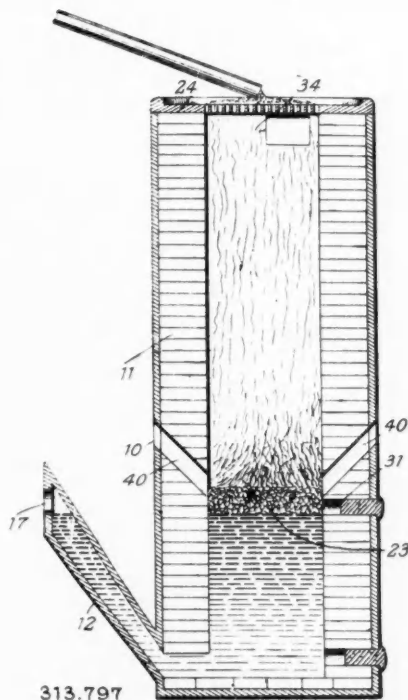
sulphate *pari passu* with its production. pH values of 3.5 to 6.5 are preferred, and by effecting the electrolysis at 48 to 50° C., crystallisation of nickel ammonium sulphate is avoided. The current density may be reduced as the concentration of nickel in the bath diminishes, and an almost complete deposition of the nickel is thus effected with small current consumption. The cathodes are preferably of aluminium or an aluminium alloy, from which the nickel is readily stripped. Residual nickel in the liquor may be recovered either as nickel ammonium sulphate crystals (obtained by concentration and cooling), or as nickel sulphide (by precipitation with ammonium sulphide).

### Platinum

A PROCESS for concentrating platinum or metals of the platinum group associated with the oxides of other metals, such as copper, nickel, or iron, is described in Specification 311,991 (Marks), dated July 24, 1928, a communication from Cayzer Tin Smelting Co. (Proprietary) Ltd., of Johannesburg, South Africa. The material is fed into a closed furnace containing a reducing atmosphere, in which the temperature is maintained at 900–1,100° C. The mixture of reduced spongy metals is removed with the platiniferous material, and the platinum is concentrated or recovered from it in any suitable manner. A rotating hearth furnace is preferably used, to enable the process to be carried out continuously.

### Purifying Molten Metals

IN Specification 313,797, dated August 1, 1928, a process and an apparatus for the purification of molten metals before casting are described by L. Jones, of Muncie, Indiana, U.S.A. The molten metal is delivered from a furnace or cupola through a perforated disc 24 into the upper end of a vessel 10 lined with refractory material 11, and falls into thin streams through the upper part of the vessel on to a layer of molten slag or



313.797

like fluxing and filtering material 23. This material floats on the molten metal which has passed through it, and its level is determined by that of a discharge port 17 in a tapering spout 12 communicating with the lower end of the vessel 10. A horizontal passage 31, normally closed, is provided in the vessel 10 for removal of spent or contaminated slag, and inclined passages 40 serve for the introduction of air or oxygen under pressure into the slag. Reagents for revivifying the spent slag may be supplied through an opening 34.

## Current Articles Worth Noting

We give below a brief index to current articles in the technical Press dealing with metallurgical subjects.

**AIRCRAFT METALLURGY.**—Metals in the construction of aircraft. H. Steudel and G. Bock. *Zeitschrift Metallkunde*, July, pp. 213-223 (in German).

Light metals in the air-cooled radial engine. F. Gossiau. *Zeitschrift Metallkunde*, July, pp. 224-227 (in German).

Materials for propellers. F. Seewald. *Zeitschrift Metallkunde*, July, pp. 227-230 (in German).

**ALLOYS.**—The electrical conductivity, corrosion and annealing properties of cadmium-zinc alloys. G. Grube and A. Burkhardt. *Zeitschrift Metallkunde*, July, pp. 231-234 (in German).

The solubility of copper in silver. M. Hansen. *Zeitschrift Metallkunde*, June, pp. 181-184 (in German). The solubility rises from 1.7 per cent. copper at 250° C. to 9 per cent. at the eutectic temperature (779° C.).

The effect of third metals on the constitution of brass alloys. I. The effect of lead. O. Bauer and M. Hansen. *Zeitschrift Metallkunde*, May, pp. 147-151; June, pp. 190-196 (in German). The ternary system Pb-Zn-Cu has been thermally and microscopically investigated in the regions 100 to 52 per cent. copper and 0 to 2.5 per cent. lead.

**ANALYSIS.**—The spectroscopic detection of lead in gold-copper-silver alloys. W. Gerlach and E. Schweitzer. *Zeitschrift anorganische Chem.*, Vol. 181, Parts 1-2, pp. 101-102 (in German).

A rapid spectro-analytic method for the quantitative determination of iridium, rhodium, and palladium in platinum. W. Gerlach and E. Schweitzer. *Zeitschrift anorganische Chem.*, Vol. 181, Parts 1-2, pp. 103-110 (in German).

**COPPER.**—The electrolytic production of copper from liquors. F. Vogel. *Metallbörse*, July 6, pp. 1489-1490 (in German).

**CORROSION.**—The action of carbonic acid under high pressure on metallic iron. E. Müller and H. Henecka. *Zeitschrift anorganische Chem.*, Vol. 181, Parts 1-2, pp. 159-171 (in German).

The determination of the resistance of Alclad to corrosion. E. Rackwitz and E. K. O. Schmidt. *Korrosion und Metallschutz*, June, pp. 130-141 (in German). Experiments on the resistance to corrosion of sheets of Alclad, duralumin, and aluminium of varying strengths in 3 per cent. sodium chloride containing 0.1 per cent. hydrogen peroxide and in a spray of 20 per cent. sodium chloride; the resistance to corrosion of Alclad under varying conditions, ranging from aluminium-coated surfaces to unprotected cut surfaces; the effect of surface damage on the corrosion-resistance of Alclad sheets, and the corrosion-resistance of duralumin in combination with pure aluminium and Alclad.

The distribution and velocity of the corrosion of metals. U. R. Evans. *Journal Franklin Inst.*, July, pp. 45-58.

A simple apparatus giving immediate and direct readings of the corrosion of metals, of the formation of incrustations, and of the oxidative power of solutions (the Oxidimeter according to Tödt). L. Köhler. *Chemiker-Zeitung*, July 20, p. 567 (in German).

**ELECTRODEPOSITION.**—Further observations on the form of electrolytically separated metals. F. Foerster and K. Klemm. *Zeitschrift Elektrochem.*, July, pp. 409-425 (in German). Gives the results of experiments on the effects of additions to the electrolytic bath on the form in which tin, cadmium and lead are deposited.

**GENERAL.**—The viscosity of solid bodies. S. Erk. *Zeitschrift Metallkunde*, July, pp. 185-189 (in German).

The preparation of aluminium alloys for microscopic investigation. H. Choulant. *Zeitschrift Metallkunde*, June, pp. 197-199 (in German). Deals with alloys of the duralumin type. Five per cent. alcoholic ferric nitrate is suggested as a new etching agent.

The resistance of chromium-nickel thermo-elements towards molten metals. R. Hase. *Zeitschrift Metall-*

*kunde*, June, pp. 200-203 (in German). Investigations on the behaviour of chromium-nickel thermo-elements in molten red brass and brass; the application of protecting tubes.

A new method in the metallurgy of nickel-copper separation. F. Vogel. *Metallbörse*, July 20, pp. 1601-1602 (in German).

**IRON AND STEEL.**—A comparison between the behaviour at the  $A_{c3}$  point of single crystal and polycrystal iron, both in the strained and unstrained state. H. Quinney. *Proc. Roy. Soc. A.*, July 1, pp. 591-603.

The response of steels at elevated temperatures. W. H. Hatfield. *Journal West of Scotland Iron and Steel Inst.*, February, pp. 60-70.

**PASSIVITY.**—The question of the passivity of metals. I. N. Stranski and Z. C. Mutaftschiew. *Zeitschrift Elektrochem.*, July, pp. 393-395 (in German).

**SAMPLING.**—The importance of sampling in the chemical analysis of alloys. Zwicker. *Chemiker-Zeitung*, July 13, pp. 546-547 (in German).

## Commercial Intelligence

The following are taken from printed reports, but we cannot be responsible for any errors that may occur.

### Mortgages and Charges

[NOTE.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debts due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case, the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.]

**ALLIED IRONFOUNDERS, LTD.**, London, E.C. Registered June 26, trust deed dated June 20, 1929, securing £780,000 debenture stock with power to issue further stock not exceeding £420,000; charged on certain specified shares, also general charge.

**DARWINS, LTD.**, Sheffield, steel merchants. Registered June 29, £60,000 mortgage, to C. T. Hennah, Richmond (Yorks), late Col. H.M. Army and another; charged on land in Sheffield.

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### Satisfactions

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**SHEEPBRIDGE COAL AND IRON CO., LTD.** Satisfaction registered July 3, £250,000, registered from December 4, 1912, to June 7, 1922.





# Monthly Metallurgical Section

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**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

## The Production and Uses of Beryllium

By Kurt Illig

(Of the Research Laboratories of the Siemens-Halske A.-G., Berlin)

The following paper was presented to a meeting of the American Electrochemical Society a short time ago. It gives an account of the work carried out at the Beryllium Research Institute, in Germany, and may be regarded as authoritative.

THE year 1928 marked the centenary of the first production of beryllium by the celebrated German investigator, Wohler. During these hundred years almost all of the alkali and alkaline earth metals such as Li, Na, K, Ca, Mg, Ba, and Sr, have been prepared from fused electrolytes in commercial quantities. In the case of beryllium alone did all of the investigators fail, until recently, to prepare this metal in such quantities as to permit a study of its properties and uses. The difficulty which stands in the way of easy isolation of beryllium is its great affinity for oxygen. Before dealing with the modern methods of production, a brief résumé of the history of this interesting metal may be worth while.

### Occurrence

Beryllium was discovered in 1798 by Vauquelin in the mineral "beryl," which had been known since antiquity. The ordinary beryl, an aluminium-beryllium double silicate, having the empirical formula  $3\text{BeO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$ , has been comparatively unknown; the more valuable varieties of this mineral, the green emerald and the blue-green to pale blue or light rose coloured aquamarine, were known in pre-biblical times.

Other minerals are known to contain more or less beryllium, as for example, chrysoberyl, also named alexandrite ( $\text{AlO}_2$ )<sub>2</sub>Be; phenacite  $\text{Be}_2\text{SiO}_4$ , which has a rather wide occurrence, and certain rare earth minerals, such as, euclase, gadolinite, helvinite, danalite, bertrandite, leucophane, melinophane—all of which are fundamentally silicates of beryllium, which differ from one another simply by the presence of silicates of other metals. A borate of beryllium, hambergite, and two phosphates, beryllonite and herderite, might also be mentioned.

### Work of Stock and Goldschmidt

The work which was decisive in the development of beryllium manufacture was accomplished by the German investigators, Stock and Goldschmidt, with the co-operation of Priess and Praetorius. They obtained metallic beryllium for the first time in the form of large reguli, directly from a fused electrolyte.

Investigations have shown that the most desirable conditions are fulfilled in general by the double fluorides of beryllium with sodium and barium. The barium-beryllium fluoride has an appreciable viscosity around 1,300 deg. C., but also has the advantage of being only slightly volatile at this temperature range; it has an appreciable dissociation within this range. The sodium-beryllium fluoride is strongly dissociated even at relatively low temperatures. This, therefore, offers a means of obtaining an electrolyte possessing a good conductivity. It has only one disadvantage, namely, that at very high temperatures the separated beryllium metal may be volatilised in appreciable quantities.

If the electrolysis is started with the sodium-beryllium fluoride and with rising temperatures some barium-beryllium fluoride is added in increasing amounts, there is obtained a useful mixture of the double salts for every temperature interval. When the final metal separating temperature of 1,300-1,350° C. is reached, it is not difficult to continue the electrolysis for many hours, provided suitable additions of both double salts are made from time to time.

### The Beryllium Research Institute

Stock and Goldschmidt protected this general method of procedure by letters patent in all large countries. They realised, however, that much additional work was necessary, before all of the details for the continuous production of large quantities of beryllium would be established. In order to study the problems on the broadest basis, the foremost German manufacturers organised a Beryllium Research Institute in 1923, under the chairmanship of the Siemens and Halske A.-G., of Berlin. It was the aim of this institute to make a systematic study of the fundamental Stock-Goldschmidt method in all its details, and to determine whether or not it was possible to produce beryllium metal in commercial quantities from fused electrolytes. The numerous variables which had to be considered made the work tedious, especially so since the temperature range of 1,300-1,350 deg. C. had never been used previously in fused electrolytic work. Some of the details of these investigations of the institute to date are herewith recorded.

### Crucible and Electrode Materials

The question of crucible and of electrode materials had to be settled first. In the early experiments of Stock and Goldschmidt, a crucible made of graphite was used as the anode. Graphite containing as little iron as possible is the only material to use. Acheson graphite is the best. The difficulties encountered in mounting the electrodes of the crucible were soon overcome. Various metals were tried as cathode; the most satisfactory was a special iron, made up into a hollow water-cooled electrode. The electrolysis current is sufficient to maintain the bath at the temperature required. At the start of the process the crucible is filled with pieces of carbon or graphite, the iron carbide is pressed firmly against these pieces, and as soon as the proper temperature is reached the pieces are quickly dumped out of the crucible and a charge of low melting sodium-beryllium fluoride is immediately put in. Then barium-beryllium fluoride is added until the required temperature range is reached for the electrolysis to begin.

Only after a large number of experiments were made was the best method for charging the crucible discovered, which method must be rigidly adhered to. Naturally there will be a relatively large burning loss of the graphite crucible, which, however, may be minimised by surrounding the crucible with another crucible packed with kryptol, to prevent contact between the very hot external wall of the graphite crucible and the air. Another method is to coat the external wall of the graphite crucible with a fused mass of the electrolyte, making an impervious coating, which is in no way objectionable since no impurities will be introduced into the electrolyte.

### First Production by Above Method

At first, relatively small reguli, of several grams only, could be separated from a single charge, and it was an exciting moment when several reguli of about 20 grams each were obtained. These reguli were still contaminated with the melt, and so were not fit for a scientific or technical investigation. Systematic and progressive development made it possible to obtain a single button of about 50 grams in weight, and at the same time containing only spots of very small amounts of

impurities. It was this material that the several laboratories connected with the Beryllium Institute, as well as others not so connected, investigated. They determined the physical properties of beryllium metal.

Hand in hand with the scientific investigations on fused electrolytes went the determination of authentic methods of analysis. The previous methods recorded in the literature were either too incomplete or entirely too circumstantial to be of undisputed value for rapid work. Very good methods are now available for the rapid determination of beryllium in the presence of aluminium and other metals. In 1926 the Institute felt convinced that in the Stock-Goldschmidt method there was a means by which a commercial production of beryllium metal might be expected. At the same time the commercial applications of this metal were studied.

#### Commercial Investigations

On the grounds of the results of the investigations of the Institute during 1926, the Siemens-Halske A.-G. decided to make a systematic study of the applications to a commercial scale. The previous work had shown that the best scientific and technical production of beryllium would be possible only if the salts used were cheaply prepared; especially if they were not hygroscopic, such as the double fluorides. The preparing of the salt from the crude mineral beryl was next investigated. Both of these problems have been solved in the past two years. Simply and cheaply prepared salts, such as the double fluorides, which also give good material yields, are being investigated now.

In the older methods the electrolyte became more and more enriched in barium fluoride, thus shortening the time of a single charge, but with the advent of the new salts it is now possible to make the process continuous and without interruption. Practically speaking, this continuous method is now limited only by the burning of the graphite crucible.

The beryllium metal is in convenient reguli of about 150 grams, varying with the size of the apparatus, and is about 98 per cent. pure. By special methods of remelting the purity can be increased to 99.9 per cent., though the yields of this very pure material are small. The main impurity of our beryllium is iron, which is introduced from the graphite crucible. The recently developed double decomposition methods of the raw beryl for the formation of the electrolyte salts produce about 65 per cent. of beryllium, calculated as metal, so that starting from the mineral the final yield of beryllium metal is about 60 per cent.

#### Occurrence of Beryl

While this work was going on, investigations were being made as to the location and size of the beryl deposits in the various parts of the world, and as to the prices at which the material was marketed. This analysis showed that as usual when the possibilities of a new use for a raw material bring it to the attention of the world, it is found in many places. Norway, Spain and especially Florida, Canada and Brazil, in the order of importance, have large deposits of beryl. There appear to be deposits larger than previously realised in south Germany, and in the Ural mountains.

It may be safely predicted that in the near future many beryl deposits will be found because now for the first time mineralogists and geologists have a stimulus for looking for this mineral. The prices for the crude beryl are exceedingly high at present, probably because there is no direct market for the material, but more probably because the owner of a beryl mine has the false impression that he has a mineral classed as a semi-precious stone. The owner must be enlightened as to the actual facts.

#### Beryllium Alloys

The most promising field which has so far been recorded in the literature is in the use of beryllium as a light metal. But the first studies of the metallographic division of Siemens-Halske have indicated that such a use is rather remote at present. Pure beryllium is an extremely hard and brittle metal, which scratches glass and can neither be rolled nor drawn because of its coarse crystal structure. Nevertheless plates of about 1 to 2 mm. by 10 to 20 mm. have been made from pure beryllium by special methods for use in X-ray work. But this is no application for which large amounts of metal need be produced.

Since beryllium has a specific gravity of 1.8 it has been considered advisable to try to alloy it with sufficient aluminium to make it workable. In this respect many interesting results may certainly be expected. It was not in the programme of Siemens-Halske to make a systematic investigation in this direction, because the realisation of our ambition to apply beryllium to arts and industry seemed only possible if beryllium alloys were developed which would compete with other alloys.

It has been shown that the addition of 2 to 2.5 per cent. of beryllium to copper or nickel produced alloys possessing properties like those of the bronzes, a fact previously unknown. In the case of additions of beryllium to aluminium the alloy showed properties somewhat like those of duralumin and silumin, though there is no advantage in favour of the beryllium-aluminium alloy. The more expensive beryllium cannot compete with silicon, magnesium, and copper in making alloys with aluminium.

Some important data on beryllium-copper and beryllium-nickel alloys might be noted. Alloys of cobalt with beryllium are very similar, but the silver-beryllium alloys have interesting properties which could not have been determined previously. In the study of beryllium-iron alloys the work has not progressed far enough to permit definite conclusions.

#### Heat Treatment

The most outstanding property of the beryllium alloys is in their improvement upon heat treatment. A copper-beryllium alloy containing 2.39 per cent. of beryllium, after ordinary quenching, has a Brinell hardness of 100 kg./sq. mm., but after heat treatment for 17 hours at 300° C. this rises to 440 kg./sq. mm. that is, to over 400 per cent. Some copper alloys containing over 6 per cent. beryllium have a hardness of over 730 Brinell numbers. These alloys cannot be worked in the untreated condition. Bronze-like properties are developed in these copper alloys, which, in the untreated condition, can be rolled, drawn or worked to the desired shape and then by heat treatment can be hardened and roughened. It is possible to make thin elastic pieces. The following information on the strength of these alloys may be of interest.

In the untreated quenched state a copper alloy containing 2.5 per cent. of beryllium has a tensile strength of 48.9 kg./sq. mm., but after heat treatment it is 135 kg./sq. mm. The differences in the transverse bending strength are most astonishing; in the untreated condition, but quenched, it is 60.7 kg./sq. mm., but after heat treatment it rises to 216.7 kg./sq. mm. In the same alloys the yield point changes from 15.7 to 128 kg./sq. mm. on heat treatment, while the elastic limit in the first case is 5 kg./sq. mm., and in the second it is 45.9 kg./sq. mm.

As the beryllium content of these copper alloys is increased the final values for these mechanical properties in the treated conditions are also increased. There is, however, a limit which is 6 to 7 per cent. Be in copper and nickel alloys. In general it may be said that the quenched alloys are very elastic and strong.

Heat treatment increases the elastic limit, and the yield point by about 800 per cent., while the tensile strength and the transverse bending strength increase about 3 to 4 fold. The ductility decreases proportionally with strength. This is not detrimental, since the shaping and working after quenching is done before it is given the heat treatment, so that ductility requirements need not be considered after the treatment. These alloys may be successfully rolled, drawn, and pressed.

#### Electrical Conductivity

It is worth noting that the electrical conductivity of these alloys is lowered rather appreciably by the addition of beryllium. Alloys of 2.5 per cent. of beryllium, after quenching at 800° C., have an electric conductivity of 9.8 recip. ohms/sq. mm., of cross section. Heat treatment changes this conductivity to 19 recip. ohms/sq. mm., and after annealing followed by a second hardening it reaches 21.5 recip. ohms/sq. mm. These alloys are more resistant to corrosion than the ordinary bronzes.

Beryllium has an interesting application as a deoxidiser in copper casting. It has been shown that the addition of 0.02 to 0.05 per cent. of beryllium to copper for casting changes the electrical conductivity of the casting from 35 to 50 recip. ohms/sq. mm. The various alloys and their applications have been protected by letters patent.

## Hardness Testing

### The Constant Size Impression Method

Of the various machines developed for the testing of hardness by the indentation method, the Brinell machine is perhaps the most popular and the most widely used, for it allows of the test being quickly and conveniently carried out, and the values obtained can be related in some measure to tensile strength. Various forms of the machine are in use, and it is customary to use hardened steel balls of 1 to 10 mm. diameter as indenter elements, machines using the 1 mm. ball having been designed for testing the hardness of small specimens and thin material. One of the chief limitations of this method of hardness testing arises from the deformation of the ball when testing hard materials.

#### The Rockwell Machine

For measuring the hardness of hardened steels and heat-treated tool steels, the Rockwell machine, where a constant load is used and the depth of penetration measured, is now quite commonly employed. With the development of extremely hard materials for use in the form of tools and dies (such as the tungsten carbide alloys, for example), the need for a satisfactory method of effecting hardness measurements is becoming more necessary. The difficulty of deformation of the indenting tool when measuring high hardness values is overcome by the use of diamond indenters in place of steel, and in various machines this material has been used.

In a recent paper by A. F. Shore read before the American Society for Steel Treating, and printed in *Heat Treating and Forging* for May, 1929, the desirability, in hardness determinations, of reading pressures required to produce a standard impression is dealt with, and the author has designed a machine known as the Monotron hardness indicator in which this principle is employed. This principle overcomes the objection which is sometimes raised against the constant load method, that with soft metals the degree of penetration is relatively large as compared with harder metals, that is, the amount of deformation which the soft metal undergoes is greater and the result is thereby affected.

#### The Monotron Method

Even with the constant size impression method the degree of penetration adopted is of importance, for it is possible that metals having similar elastic limit hardnesses will give very different results with greater penetration if they behave differently under extreme cold working. The errors in measuring high hardness values due to deformation of the steel ball, as in the Brinell machine, are overcome to some extent by the use of a diamond pointed indenter in the Monotron machine.

With a machine embodying the constant size and varying pressure principle, one of the chief difficulties in the way of obtaining accurate measurements of the depth of penetration is due to the elastic shortening of the penetrator element when under load. It is not practicable to make allowances for this factor for different hardness values and in the Monotron machine the depth micrometer is held in a compensator, and so given a retractile movement equal to the elastic shortening of the diamond point when under pressure. The load required to produce a standard penetration, and the depth of the penetration, are read off on two dials which form part of the instrument. For universal application a 0.75 mm. diamond ball point is used, the standard depth of penetration being 0.0018 in., which is 6 per cent. of the ball diameter. With this penetrator the entire hardness range encountered in metals from lead to tungsten carbide alloys can be satisfactorily measured.

#### Method of Making the Test

No special difficulty attaches to the carrying out of the test, irregular shaped pieces of metal being satisfactorily accommodated by a tilting table which can be replaced by a V-block for cylindrical specimens. The values read off on the dial recording pressure are in kilograms and pounds, but other scales can be substituted such as are giving diamond ball Brinell values direct. The modulus of elasticity of the material under test does not affect the result since the depth of the penetration, or hardness value, is read while the load is applied. A protecting cap for the diamond point acts as

a contact for operating the depth gauge, and since the latter moves relative to the position of the cap it has the advantage that any yielding (say of a hollow piece under test) does not affect the result. The depth scale is divided into nine divisions each representing 0.0002 in. or 0.005 mm.

#### Determinations on Very Hard Products

As already indicated, the development of extremely hard metallurgical products has led to increasingly exacting demands being made on hardness testing machines. Of these, surfaces hardened by the nitriding process and tungsten carbide alloys may be cited, the hardness of some of the alloys in this class being twice as much as that of quenched high-carbon steels. Such materials are well beyond the range of hardness which can be satisfactorily determined by hardened steel balls or indenters, and only carefully selected diamonds can be used. Even these must be used with caution on the harder grades of tungsten carbide alloys. The Monotron instrument with its spherical diamond indenter, which gives a straight increment line, can be used to advantage in determining the hardness of these extremely hard products.

In testing materials of this kind, it is advisable to use a depth of penetration corresponding to three divisions only, which does not impose an unduly severe strain on the diamond and yet is quite satisfactory for the purpose. With hardened steel a penetration equal to three divisions requires a pressure of from 35 to 38 kilograms, which when multiplied by three gives the value needed for the standard penetration of nine divisions or 0.0018 in. With superhard steels this value to yield the standard penetration might be as high as 120. With nitrided steel it is of the order of 117, or 39 kilograms for a penetration equal to three divisions.

Values on tungsten carbide alloys vary considerably, one of the softer but tough grades recording 45 kilograms with a depth of three divisions or 135 when calculated to the standard, a harder and more brittle sample yielding corresponding figures of 65 to 75 and 195 to 225 kilograms. On hardened steel, the diamond will last for thousands of tests taken to the full depth of nine divisions, but is not capable of being used on tungsten carbide to this depth.—M. C.

### Estimating Iron in Chromium Alloys

THE use of alloys containing iron and chromium, and often also nickel, is at the present time becoming very widespread in engineering, and therefore the following details (supplied by a correspondent) may be of interest to those engaged in the analysis of these alloys. The usual method of separation of iron from chromium is by means of sodium peroxide, but the author has found that the following method gives quite accurate and reliable results on this class of material. The method is based on the oxidation of the chromium by ammonium persulphate and then precipitation of the iron as usual with ammonia. The estimation is carried out as follows:—

From one to three grams of sample is taken (depending on the amount of iron suspected) and dissolved in dilute sulphuric acid if possible. If, however, the alloy is not completely soluble in sulphuric acid, then hydrochloric acid or aqua regia must be used to dissolve it, and then sulphuric acid must be added and the solution evaporated until white fumes are evolved. The solution is then diluted with hot water and boiled. If any silicon is precipitated at this stage it should be filtered off. The solution is now cooled, diluted to 250 ccs., and a measured quantity taken so as to give approximately 0.1 to 0.2 g. of iron in the estimation (a larger quantity may be difficult to filter). This is then made up to about 150 c.c. bulk, about 2 g. of ammonium persulphate crystals added, and the whole boiled for about a quarter of an hour. All the chromium in the alloy will now be oxidised, and any manganese will be precipitated as manganese dioxide, which should be filtered off. Ammonium chloride and ammonium hydrate are now added to the solution to precipitate the iron, which is filtered off, after boiling, and ignited and weighed as ferric oxide as usual. The percentage of iron is then calculated from the weight of sample taken as usual.

The resulting filtrate may sometimes be used for the estimation of nickel in the alloy by the dimethylglyoxime process, but here the chemist must use his discretion, as this depends on the amount of nickel present in the alloy.

## Metallurgical Topics: Monthly Notes and Comments

### From Our Own Correspondents

#### Some Alloys of Aluminium

WITH regard to an article on "Some Alloys of Aluminium," which appeared in this journal last month, Lightalloys, Ltd., of Alpax Works, St. Leonards Road, Willesden Junction, London, write as follows:—

"We notice that in the second column of the article under the heading 'Nickel in Aluminium Bronzes,' there are a number of rather misleading statements. Firstly, it is stated that the addition of nickel to the aluminium bronzes described leads to the formation of a harder and better alloy. 'Aluminium Bronzes' is a term which is usually applied to copper alloys containing a small percentage of aluminium, as for instance, 92 per cent. copper, 8 per cent. aluminium, and not to the high aluminium alloys as described in the previous part of the article; in fact, the whole of the alloys described in the paper are essentially aluminium alloys, and in no wise can be described as aluminium bronzes when that term is used in the popular sense.

"Further, it is stated that among the aluminium bronzes there are three which stand out above all the others, viz., 'Alpax,' 'Lynite' and 'Y' alloy. These three alloys are essentially high aluminium alloys. 'Alpax' certainly derives its name from that of the inventor, Aladar Pacz, but is a silicon aluminium alloy which has been 'modified' by one of the various patented processes. Ordinary aluminium silicon alloy is not 'Alpax,' this term being a trade name for the modified alloy.

"With regard to the strength values given for 'Alpax,' the figure for the alloy described as 'melting under a flux containing sodium fluoride,' is very much lower than is now obtained in ordinary works practice."

#### The Reply

THE author, to whom the above comments have been submitted, replies as follows:—

"In the first place Messrs. Lightalloys object to the use of the term 'aluminium bronze' for alloys which are not composed of a preponderance of copper. This is purely a matter of taste; the term 'bronze' itself signifies an alloy of tin and copper (with or without other additions), and it so happens that with these two metals the alloys which are of most value are those which contain a high percentage of copper. When, however, the term 'bronze' is transferred to aluminium and copper, it is found that both the alloys containing much copper and those containing little copper are very valuable commercially; the trade custom of calling only those alloys high in copper 'aluminium bronzes' is only an artificial distinction. Your correspondents themselves state that these alloys 'can in no wise be described as aluminium bronzes when that term is used in a popular sense.' Articles written for THE CHEMICAL AGE are definitely 'scientific' in contradistinction to 'popular.'

"With reference to the alloy Alpax, I have re-read the section that I wrote on this alloy and must admit that I have not been as clear as could be desired, although I was quite aware that Alpax is a 'modified' aluminium silicon alloy. I have not, however, stated, as your correspondent implies, that ordinary aluminium silicon alloy is Alpax; in fact, I have stated that Alpax is an aluminium silicon alloy the strength of which can be increased (owing to the diminution of silicon grain size) by fusion under a flux containing sodium fluoride. Surely this does not imply that any aluminium silicon alloy is Alpax.

#### Strength of the Alloys

"Further, concerning the strength of these alloys, I would point out the enormous difference that exists between the tensile strength as shown on a special test piece and that shown on a section cut from a casting. With reference to the alloy under discussion I would like to quote the figures of Corson, the acknowledged international authority on the subject, from his treatise on *Aluminium and Its Alloys*. He gives as the tensile strength 30,000 on the separate test piece, but only 19,800 on cast metal. These figures are from an Alpax containing 13 per cent. of silicon and which has been modified by melting under a fluoride flux. Anderson in the *Metallurgy of Aluminium and Its Alloys* gives similar figures. I do not

doubt that the special alloys of Messrs. Lightalloys, Ltd., will often test at considerably above the figures given above when tested on a test piece; but I can state categorically that alloys cast in our own foundry at Loughborough very seldom exceed this figure until after heat treatment."

#### Lead Smelting

THE first of a series of papers to be issued on "Smelting in the Lead Blast Furnace," by G. L. Oldright and V. Miller, has appeared as the August Serial (No. 2,954) of the Bureau of Mines, Washington. It deals with a method for approximating the form of the lead in slag and other products of the lead blast furnace. In the lead blast furnace, as in the blast furnace smelting the iron ores, coke economy is essential and the operator tries not to use more coke than is necessary to metallise the lead in the charge and to separate it from the slag-forming constituents. Over-reduction is contra-indicated, not on grounds of economy alone, but because it leads to accretions of metallic iron, or Speiss, in the crucible, and seriously hampers working. To help in directing aright the operations involved, the forms in which lead may be present in the slags have been ascertained, more particularly as regards its occurrence as metal, or as sulphide, or as silicates, or mixtures of these. These three forms exhaust those in which lead is found to be present in lead blast furnace slags. By treating lead sulphide with brine containing ferric chloride it was found possible to separate it completely from mixtures, while lead silicate can easily be prepared artificially by means which are described and serve to show the nature of reactions to be avoided in actual smelting operations. Methods of analysis are given, and the paper is one which lead smelters should find both useful and suggestive.

#### The Diffusion of Gases through Metals

IN the July issue of the *Revue de Métallurgie*, V. Lombard gives the results of some interesting experiments he has made on the diffusion of gases through metal plates, used as partitions. The gases used were hydrogen, helium and argon, and the metals were nickel, iron and platinum, from which it may be seen that the research was not of an academic nature, but one from which practical results of use in aeronautical engineering and in electric light bulb technology, may be adduced. He has brought out the following facts: (1) The rate of diffusion of hydrogen through nickel plates (those employed varied from 0.160 to 2 millimetres in thickness) does not, provided the temperature be constant, vary in functions of the time, although the amount passed varies a little, according, probably, to the difference in structure of the nickel. The permeability of nickel by hydrogen is not affected by the presence of moisture, or by that of small amounts of oxygen, such as may occur in the hydrogen of commerce; (2) When an inert gas is present the hydrogen passes through the nickel at a rate proportional to the square root of its own pressure, and the total pressure of the mixture exerts no influence on the result. The permeability of nickel to nitrogen and to argon, although small, is not nil, whereas helium permeates it more rapidly than hydrogen. The results with platinum and with iron plates are to be published later.

#### Cementation as a Corrosion Protective

THE fourth, and final, part of the report of the French Committee on the Corrosion of Light and Ultra-Light Alloys, by J. Cournot and E. Perot, deals with cementation methods of protection. The first section is devoted to the influence of the cementation of aluminium by copper; the second deals with the cementation of aluminium and duralumin by various ferro alloys. The process employed was: (1) Direct, by cementing in an electric furnace with the metal itself, and (2) By cementing in a bath of appropriate salts. In the first case adherence of the coating was difficult to secure; in the second, the presence of saline matters may become injurious to the surface of the coated metal. At the temperature of the experiments it was not found possible to cement aluminium by means of ferro-alloys, such as ferro-chrome, etc. On the other hand, in the case of duralumin, "an entectic is precipitated throughout the mass of the metal," due, the authors think, to the expulsion of the copper in the duralumin, and

its combination with some of the aluminium itself. A final section of the research deals with cadmium plating on aluminium and duralumin, and with chromium plating over the cadmium. Indeed, the metals subject to the experiments appear to have first been copper-plated; then cadmium-plated, and, finally, to have been again plated with the chromium, a method which is said to have given very satisfactory results. Samples so treated bent well, without peeling, and even stoop drawing, without revealing any signs of cracking so far as the surface platings were concerned.

### **Pure Iron**

ALL that is known, and has hitherto been put on record with reference to the physical, thermal, magnetic and electrical properties of pure iron, will be found summarised and compiled from the wide literature of the subject in Bulletin 296 of the Bureau of Mines. This is but a section of the contents of this admirable publication, which is by Oliver C. Ralston, and ostensibly deals only with the subject of its official title "Iron Oxide Reduction Equilibria." Its secondary title amplifies this somewhat, being a "A Critique from the Standpoint of the Phase Rule and Thermodynamics." In point of fact, it contains a good deal more than its title implies and is a most exhaustive summary not only of its subject but of many incidental and accessory aspects of pure iron and iron oxide metallurgy.

The main headings of the sections indicate the scope of the work; they are: Ferric Oxide; Higher Oxides of Iron; The System Ferric-Oxide Magnetite; Magnetite, Iron-Oxygen Intermediate Complexes; Ferrous Oxide; Iron-Oxygen Complexes; The System Iron; and The System Iron-Carbon. The work will be valued, as the matter it contains, conveniently grouped and compiled, would otherwise have to be pursued through countless transactions of innumerable societies and institutions. Yet the data given are precisely those of which the iron metallurgist is most often in need, and usually in doubt as to where to find them.

### **Steel "Failures" and Margins of Safety**

THE failure of the bolts in an aeroplane disaster that cannot as yet be regarded as ancient history has given engineers and metallurgists, alike, furiously to think. That one bolt should fail is regrettable, but, in existing circumstances, not unnatural. That a second should fail is bad. That, with extra duty, one or two more failed likewise, could, probably, be anticipated. The trouble is that it was not anticipated. The margin of safety seemed ample, but certain percentages of failure, or rejection of a given lot of bolts for use in circumstances of intensely severe stress, which may at any time become wholly exceptional, should ensure the rejection of the lot. Alternatively, as a contemporary rightly suggests, a factor of safety might be adopted, based not on strength or service stresses but on time of service or endurance. That is, after a length of service bearing some proportion to the ascertained fatigue strength of the more severely stressed part or parts, the whole system should be overhauled and such parts replaced. This, of course, still leaves open the question of occasional sudden and unexplained failure, of the kind that no preliminary testing or method of inspection can at present guard against.

### **"Natural" Chromium-Nickel Steels**

FOR certain parts of certain machines, expense is no object. The best and strongest material alone should be used; "strong," that is, in respect not of any generalised or theoretical stresses, but in resisting just those special, extraordinary and cumulative stresses the part is called upon to bear in practice. This is, of course, the merest truism. Nevertheless, it is readily conceivable that material better than that habitually employed should, if possible, be used, and the resources of metallurgy are surely equal to making a material that will stand up to anything—for a time—and of replacing before that time has elapsed. A description was given by Mr. Arthur Richards, more than twenty years ago, of an extraordinarily shock-stress-resistant steel made, somewhat fortuitously, at Bolckow Vaughan's works, Middlesbrough. It was made from pig iron that had itself been made from a Greek ore containing notable percentages of chromium and nickel. It was difficult to smelt, and very refractory, and the resulting pig iron contained 1.750 per cent. of nickel and cobalt, 4 per cent. of chromium, and 0.4 per cent. of silicon. It

was blown in an acid Bessemer converter and made an exceptionally tough steel with 1.4 per cent. of nickel, 0.25 per cent. of cobalt, and 0.32 per cent. of chromium. On a Stead-Richards machine such steel withstood 210,910 revolutions to fracture, as against 77,33 for a 0.46 carbon steel, and 100,175 in the case of a silicon steel (carbon, 0.45; silicon, 0.24). Later on the Mayari ores of Cuba, which contain similar constituents, were successfully employed to produce a very similar steel, with rather more chromium, which likewise gave remarkable results. In the case of monel metal, what is called a "natural alloy" has been shown to be superior to one deliberately made to as nearly the same composition as possible. Automobile and aeronautical engineers and metallurgists might be well advised to test the virtues of a "natural steel" alloy of the kind described, and if it be not now procurable there is no reason why it should not be made, seeing that ores which contain the necessary constituents in the necessary quantities are fairly abundant.

### **Steel Strip and Bar**

WE have received from United Strip and Bar Mills, Ltd., Sheffield, a handy pocket-size booklet setting out the various sections of steel strip and bar they have available for their customers' use. Many useful tables are also incorporated. The United Strip and Bar Mills, Ltd. (which is a branch of The United Steel Companies, Ltd.) caters specially for the reinforced concrete trade. The company has a service department whereby reinforcement bar is supplied in lengths, ready bent, hooked, marked and bundled, and delivered to suit the contract, thus relieving the contractor of an arduous duty on site. A special department under a ferro-concrete engineer will also get out quantities and advise generally on ferro-concrete construction for the needs of the smaller contractor. Steel users in the South are catered for by the large stocks for immediate delivery held at the London warehouse. Those interested may obtain a copy of this useful booklet on application to the Publicity Department, United Strip and Bar Mills, Ltd., The Ickles, Sheffield.

### **A New Technical College**

THE Constantine Technical College, Middlesbrough, which the Prince of Wales is to open officially next spring, will be opened for students on September 16, when courses for day and evening students will begin. The college has cost £80,000 to build and £45,000 to equip, and is in the charge of Dr. D. H. Ingall, who anticipates that between 700 and 800 students will be enrolled. Special attention is being paid to the industrial needs of the district, and the college is certain to play an important part in the supply of highly trained technical and semi-technical workers needed in the great industries of Tees-side. The courses include one for iron and steel operatives, which has been designed for those who, while not desiring to take the higher courses in metallurgy, can secure a certain amount of scientific training. The equipment of the college, towards the cost of which substantial donations have been made by the principal firms in the district, includes a high frequency electric melting furnace and the largest experimental rolling mill installed in any technical school in the country.

### **Abnormality in Case-Carburised Steel**

DIFFERENT results on depth of carbon penetration, grain size and uniformity of surface hardness are often found in carburised steels of essentially the same chemical analysis, and the term "abnormality of case-carburised steel" has been used to denote certain of these variations.

A study of this perplexing problem is being conducted at the Pittsburg Experiment Station of the United States Bureau of Mines, Department of Commerce. It has been found that steels may show two distinct types of abnormality on carburisation; grain size abnormality and structural abnormality. The first is usually due to the addition of certain elements to the steel and may be regulated by the elements added in the manufacture of the steel. Structural abnormality has been found to be associated with either a high FeO content or with the presence of very small inclusions in the experimental steels made by the Bureau of Mines. Apparently as the inclusions become larger they affect structural abnormality less and less. Heat treatments do not affect this type of abnormality.

## Trade, Commerce, Finance: The Month in Review

### From Our Market Correspondent

AUGUST has maintained its reputation as the holiday month, and the iron and steel trade during that period has been particularly quiet. The annual closing down of works for holidays or repairs or both has been general throughout the country, and both production and consumption have been materially curtailed. The reduction in the volume of business has been gradual over several weeks, and as the mills were executing orders more quickly than new ones were coming in, the stoppage of ten days or a fortnight gave a welcome opportunity of accumulating orders so as to give a good start when the autumn revival in trade sets in.

#### Improving Prospects

There is nothing abnormal in this position. It is the usual experience year after year, and the steel makers are prepared for it. With the advent of September a change for the better may be expected, and already there are signs that business is becoming more brisk. The general expectation is that the last quarter of the year will prove to be quite good for the steel works. Naturally during the past few weeks there has been very little buying, as most consumers had covered themselves for their requirements for a month or two ahead and the holiday season does not stimulate fresh buying, but the feeling is that by the middle of September there will be a considerable amount of new business transacted.

It is too much to expect that all the steel works will be fully employed during the closing months of the year. That is a condition which may be hoped for but which is extremely unlikely to be attained in the present stage of development of the iron and steel industry. So far, there have been no really effective steps taken to bring productive capacity into economic relation with the demand, and until that is done there is no alternative but to have a number of plants working part time. That is the prospect for the great majority of the works, and it is the problem which will have to be solved before prosperity can return to the industry as a whole. The steel makers are fully alive to this fact, and in some of the recent amalgamations action is already being taken along this line.

#### Rationalisation of Orders

Where within the control of one group there are several plants capable of turning out the same class of work, it is far better that the available orders should be so distributed that some of these plants are working full time, even if others are closed down altogether, rather than having all of them working intermittently. This policy is bound to involve hardship not only on the workmen but on the staff; yet it is the only way to stop or at least to minimise the ridiculous cutting of prices which is being carried on by some of the works who in their anxiety to divert orders to themselves appear to have lost their sense of business values. Special qualities of steel which rightly command an extra price and for which the customer has always been willing to pay this extra are being offered at practically the same price as ordinary steel; boiler plates are being sold at very little more than ship plates; and so the ground is being cut from under the feet of those makers who have devoted their energies to the production of the highest quality of steel.

It is always easy to get orders by reducing prices, but it is extremely difficult to restore prices to their former level. Definite instances could be given of reductions of 10s., 20s., and even 25s. per ton in the prices of special steels, with the sole object of filling the order book, with the result that what had previously been a legitimate profit to one works disappears or is turned into a loss to the other makers who have needlessly cut the price. As already stated, this kind of thing will only cease when the offenders have a sufficiently good order book to remove the necessity of poaching on the grounds of their competitors.

#### Amalgamations

The latest amalgamation in the steel trade is that of Dorman, Long and Co. and Bolckow Vaughan and Co. on the North East coast. Rumours of it had been prevalent for some time. The amalgamation is a natural one. The undertakings are situated fairly close together and their activities are similar in many respects. The fusion should offer good scope for the avoidance of overlapping, the cutting out of superfluous plant

so as to ensure the most favourable concentration of production, and the simplification of administration. This process of amalgamation or absorption is going steadily on.

Another old established steel works, with one of the most honoured names in the history of the industry, is about to disappear. The works of Henry Bessemer and Co., Ltd., of Sheffield, have been taken over by another local firm of wheel and axle makers, and they will shortly be closed down for good. After a long and successful career they, like so many others, fell on evil times, and for the past few years have been struggling to keep going. Now their end has come. It is all part of the process of reorganisation of the steel industry which can be no longer delayed.

#### Expenditure on Plant

Another sign of this reorganisation is the expenditure on new plant which is being incurred by many of the works. Considerable sums are being expended with the object of installing the latest methods of production so as to reduce the cost. Selling prices move upwards very slowly, nothing like fast enough to keep pace with the advance in prices of raw materials, and the steel makers are again forced to concentrate on the reduction of manufacturing costs so as to maintain some margin between cost and selling price. The time and skill which have been devoted to the improvement of steel works practice have opened up a field of possibilities for the steel maker, and there ought to be no hesitation in taking advantage of them when once the difficulty of finding the necessary capital has been overcome.

#### General Market Conditions

There is not much to be said about general market conditions during the past month. The quietness has been common to all sections, although the pig iron market has been the least affected. The output from the blast furnaces has been fully absorbed, and still the demand has not been completely satisfied. The production of iron during the stoppages of the steel works has helped in overtaking arrears. There has been no sign of any weakening in prices. The recent advances have been firmly maintained, and a further upward move is not unlikely as soon as the works get into full swing again.

The blast furnace owners are expecting a good demand for iron on forward contracts and they will probably seek to cover themselves against probable increase in cost. The price of blast furnace coke is still steadily moving upwards, and this movement is not likely to be checked by the increase in output which it is hoped to achieve. The hematite makers also are predicting higher prices in the near future, as the present prices show no margin of profit. It is not possible to forecast any move in steel prices.

The official prices for the controlled products remain unchanged, while for the uncontrolled steels, such as boiler plates and small bars, there has been a slight weakening. Boiler plates have been sacrificed in order to secure contracts for tank plates, and small bars are meeting with more severe competition from the Continent. Nevertheless, the steel makers are likely to be faced with increased costs in fuel and scrap and it would therefore be only reasonable if selling prices were suitably adjusted. It would not be surprising if the associated steel makers decided to ask a further advance for steel plates and sections, but the competition for the uncontrolled materials is so keen that advances can hardly be expected.

The output of pig iron for July was 671,900 tons compared with 657,800 tons in June, there being two more furnaces in blast. The output of steel was 804,800 tons compared with 830,900 tons in June.

#### "Aluminium Facts and Figures"

A FURTHER series of data sheets for inclusion in their handbook, "Aluminium Facts and Figures," has been issued by the British Aluminium Co. A.73 and A.74 show enlarged tables of the properties of stranded aluminium and steel-cored aluminium conductors, revised in conjunction with the British Engineering Standards Association. A.166 is a new data sheet on expanded aluminium sheets. The metal in this form makes up into economical framing, backgrounds and screening, and a variety of light structural uses. Further data will be issued from time to time.

## Some Inventions of the Month

## By Our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature, published weekly in THE CHEMICAL AGE.

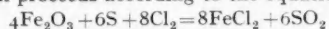
## Alloys

AN alloy suitable for electric cable sheathings and other articles comprises lead with 0.02-0.1 per cent. of calcium, the product being heated to 310-325° C., quenched, and aged. The article may be made from the alloy either before or after the heating. See Patent Application 314,522, bearing the International Convention date June 29, 1928, by Standard Telephones and Cables, Ltd., of London, Assignees of R. S. Dean, of Riverside, Illinois, U.S.A.

## Extracting Metals

ZINC, iron, and other metals are extracted from their ores by treatment with acid on the countercurrent principle, purification of the resulting solutions by means of the sulphides of the respective metals, and recovery of the metals by electrolysis. The purification is preferably effected by aspirating the solution through a cake of the sulphide. A process and an apparatus for the extraction of zinc by sulphuric acid from roasted or oxidic ores, with secondary recovery of copper, cadmium, lead, silver, arsenic, antimony, nickel, and cobalt, are described and illustrated in detail. See Patent Application 313,858, bearing the International Convention date June 15, 1928, by L. Sturbelle, of Brussels.

In a process for the extraction of iron from oxide ores by chlorination, described in Specification 316,404, dated July 3, 1928, by W. S. Millar, of London, the ore is mixed with sulphur or sulphide ore or material such as pyrites, chalcopryrite, pyrrhotite, mattes, spent oxide, etc., and the mixture is heated with chlorine or a gas containing free or combined chlorine, such as sulphur chlorides or volatilised ferric chloride. In the simplest case, using sulphur and chlorine, the reaction proceeds according to the equation



Non-ferrous metals present are also converted into chlorides, and the chlorine may be recovered from the various metallic chlorides by electrolysis or oxidation and used for the treatment of a further quantity of the ore-sulphur or ore-sulphide mixture. Two methods of separation of the non-ferrous metals from the iron are described in detail, one being based on the separation of ferrous chloride by crystallisation from a concentrated aqueous solution of the product from the chlorination, and the other on the separation by volatilisation of ferric chloride formed by subjecting the product from the chlorination to the oxidising action of chlorine or of limited quantities of air or oxygen at a moderate temperature. The chlorination reaction is strongly exothermic and the desired temperature is maintained by regulating the rates of admission of the reagents.

## Iron

A PROCESS and apparatus for reducing iron ores and utilising the hot products of the reduction for cracking oils are described and illustrated in detail in Patent Application 313,597, bearing the International Convention date June 16, 1928, by Trent Process Corporation of New York, U.S.A. A mixture of ore and carbonaceous material is heated in a furnace, and the iron produced is deposited in oil which thus protects it from oxidation, while the oil vapours rising in the furnace are cracked, by contact with the hot reduced ore, and the products of the cracking are removed. The ore may in some cases be used without the carbonaceous material, the reduction being effected by the oil vapours.

## Iron Alloys

RUSTLESS or stainless iron alloys of a carbon content not exceeding 0.1 per cent. are obtained by a process described in Specification 315,444, dated February 14, 1928, by L. F. Reinartz and J. H. Nead, of Middletown, Ohio, U.S.A. A bath of molten iron, of very low carbon content and very low impurity range under oxidising conditions, is prepared in an electric furnace, preferably by diversion of molten ingot iron from an open hearth furnace. The molten iron is then degasified in the electric furnace, and a reducing slag is formed on the surface of the "killed" iron, whereupon an alloying metal, substantially free from carbon, is added while maintaining the temperature of the furnace. Specified alloying metals are chromium, nickel, vanadium, cobalt, molybdenum, and titanium.

ALLOYS of iron with chromium, manganese, tungsten, or the like, having a ratio of alloying metal to carbon below 30, are decarburised with little or no external heating by subjecting them to a blast enriched to contain not less than 48 per cent. of oxygen so that a temperature of at least 1,600° C. is maintained by oxidation of the constituents of the alloy, the blast being applied to the surface of the molten alloy so as to protect the linings of the containing vessel from the intense heat, and the blowing being continued until the ratio of alloying metal to carbon is above 30. The final decarburisation to the desired limit may be effected by blowing with hydrogen to effect the elimination of the carbon with relatively small loss of alloying metal. The hydrogen is preferably introduced beneath the surface of the molten metal. See Spec. 316,329 (Potts), dated March 27, 1928, a communication from Electro Metallurgical Co., New York.

## Platinum

A PROCESS for the recovery of metals of the platinum group from ores, concentrates, mattes, or residues containing them, especially ores in which they are associated with sulphides of nickel, copper, or iron, is described in Specification 316,063, dated May 1, 1928, by A. R. Powell, of Amersham, Bucks, E. C. Deering, of Barnet, Herts, and Johnson Matthey and Co., Ltd., of London. A matte containing the precious metals is first formed by smelting the ore, etc., and this matte is treated to form or provide within it a proportion of free metal of the iron group, especially iron and nickel, which acts as a collector for the precious metals. A disintegrating agent, such as an alkali hydroxide, carbonate, or sulphide, is added to the molten matte, from which, on cooling, the metal crystallises out. The cold matte is crushed and the meta is separated from the sulphides by magnetic or mechanical means.

## Separating Ores

A PROCESS for facilitating the recovery of oxidised ores, especially of copper, in their separation by froth flotation methods is described in Specification 314,822, dated March 3, 1928, by R. J. Lemmon, of London. The ore particles are treated, during or prior to the froth flotation, with a small proportion of a thiocyanogen compound. Specified compounds are esters, salts, or other derivatives of isothiocyanic acid, decomposition products formed by treating a cyanogen sulphide, or an isomer or alkyl derivative thereof, with alkalies and isopersulphocyanic or isodithiocyanic acid or their soluble salts or esters. Such compounds may be used either alone or in admixture with additional sulphur.

## Treatment of Ores

OXIDE ores, particularly of tin, and superficially oxidised sulphide ores, are subjected to a superficial reduction preliminary to separation by flotation. The reducing agent may be used in solution, or hydrogen or a gas containing it may be added or generated in the flotation apparatus. See Patent Application 311,239, bearing the International Convention date May 7, 1928, by J. Barnitzke, of Clausthal, Harz, Germany.

A METHOD of extracting zinc and like volatile metals from oxidised ores is described in Patent Application 312,667, bearing the International Convention date May 30, 1928, by A. Folliet and N. Sainderichin, of Paris. A current of air, heated to 650 to 800° C., is blown over a charge comprising a mixture of fine ore with coal or lignite and a small quantity of an alkali chloride, e.g., in the form of sea water. In the preferred arrangement the air is delivered through a tangential nozzle at the lower end of an inclined rotary furnace having communicating compartments, and the volatile metal is discharged at the top to a condenser, filter, etc.

ORES or metallurgical products containing metals capable of conversion into carbonyls are rendered amenable to the action of carbon monoxide to effect such conversion by removing or rendering innocuous any easily fusible or slag-forming substances such as zinc, cadmium, tin, bismuth, lead, or silicic acid combined with alkalies, etc. Zinc or cadmium may be removed by volatilisation in an inert or reducing atmosphere. A material containing iron and zinc may be oxidised and thereafter treated with hydrogen at 475° C. to reduce the iron, but not the zinc. Slag-forming substances may be removed by flotation or by magnetic separation. See Patent Application 312,629, bearing the International Convention date May 29, 1928, by I.G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany.

## Current Articles Worth Noting

We give below a brief index to current articles in the technical Press dealing with metallurgical subjects.

**ALLOYS.**—The structure of gold-nickel alloys. W. Heike and H. Kessner. *Zeitschrift anorganische Chem.*, Vol. 182, Part 3, pp. 272-280 (in German).

Investigations on the stability of the hysteresis of iron-nickel alloys. G. Gossels. *Zeitschrift anorganische Chem.*, Vol. 182, Part 3, pp. 19-27 (in German).

Germanium. XXXI. Alloys of germanium: silver-germanium. T. R. Briggs, R. O. McDuffie and L. H. Willisford. *Journal Physical Chem.*, July, pp. 1080-1096.

**COPPER.**—The recrystallisation of electrolytic copper after hot rolling. A. Tafel. *Zeitschrift Metallkunde*, August, pp. 265-267 (in German).

**CORROSION.**—The effect of various salts on the dissolution of pure aluminium in hydrochloric acid. J. Calvet. *Comptes Rendus*, July 22, pp. 183-188 (in French).

The corrosiveness of soils with respect to iron and steel. H. D. Holler. *Industrial and Engineering Chem.*, August, pp. 750-755. A method of studying soil corrosion based on the relation of salt content, acidity, and corrosiveness of soil to rainfall is suggested. The corrosiveness of soils as indicated by the initial losses of buried specimens in humid areas of the United States has been correlated with their acidity.

The artificial corrosion of Japanese special alloys. D. Uno. *Korrosion und Metallschutz*, June, pp. 122-130; July, pp. 147-156 (in German).

**ELECTRODEPOSITION.**—Electrolytic deposits on aluminium and its alloys. H. K. Work. *Revue de Métallurgie*, July, pp. 378-390 (in French).

**ELECTROMETALLURGY.**—The electrolytic production of metals: To-day's methods and their practical application. G. Eger. *Chemische Fabrik*, July 10, pp. 323-324; July 17, pp. 333-335; July 31, pp. 351-352 (in German). Deals with the production of copper, the noble metals, zinc, cadmium, lead, tin, nickel, cobalt and iron; and also with chromium and cadmium plating.

The separation of bismuth by rapid electrolysis in acid liquor. A. Jilek and J. Lukas. *Collection Czechoslovak Chemical Communications*, July, pp. 369-376 (in French).

Advances in the domain of zinc electrolysis with especial regard to the Tainton process. G. Eger. *Metall und Erz*, Vol. XXVI, Part 15, pp. 373-383 (in German). The development of zinc sulphate electrolysis according to the processes used hitherto; the more important fundamentals of the process, especially the effects of changing acid content and changing current density; the chief characteristics of the Tainton process—strongly acid solutions in lixiviating and high current density; an account of the great installation at Kellogg, Idaho.

**FATIGUE.**—High frequency fatigue. C. F. Jenkin and G. D. Lehmann. *Proc. Roy. Soc. A*, August, pp. 83-119. The object of the researches described was to determine the effect of frequency of alternation of stress on the fatigue limits of various metals. Tests at frequencies up to 20,000 periods per second were made on rolled, normalised and hardened steel; rolled aluminium; annealed copper and normalised Armco iron.

**GENERAL.**—The content of hydrogen and carbon monoxide of some metals melted *in vacuo*. A. Villachon and G. Chaudron. *Comptes Rendus*, August 12, pp. 324-326 (in French).

The colouring of metals. I. and II. H. Kurrein. *Chemiker-Zeitung*, August 7, pp. 609-610; August 14, pp. 630-631 (in German).

Bimetals. W. Rohn. *Zeitschrift Metallkunde*, August, pp. 259-264 (in German). Bimetals—i.e., strips formed of two metallic layers of different coefficient of expansion.

The solubility of gases in melts of pure aluminium and of an aluminium alloy. W. Claus, with S. Briese-meister and E. Kalaehne. *Zeitschrift Metallkunde*, August, pp. 267-268 (in German). Investigations on the solubility, in melts of pure aluminium and an American alloy (8 per cent. copper and 92 per cent. aluminium), of nitrogen, oxygen, carbon monoxide and dioxide, sulphur dioxide, hydrogen, illuminating gas, methane, ethylene and water vapour; a note on Y alloy.

Investigations on the diffusion of gases through metals. V. Lombard. *Revue de Métallurgie*, July, pp. 343-350 (in French). Deals with hydrogen-nickel; iron, platinum-hydrogen; and nickel-nitrogen, argon, helium.

A high-temperature modification of manganese. E. Persson and E. Ohman. *Nature*, August 31, pp. 333-334.

**IRON.**—Investigations of equilibria in the reduction, oxidation, and carburisation of iron. VIII. R. Schenck, T. Dingmann, P. H. Kirscht und H. Wesselkock. *Zeitschrift anorganische Chem.*, Vol. 182, Part 3, pp. 97-117 (in German). The system iron-oxygen.

**STEEL.**—Elastic limit steel. F. G. Martin. *Journal West of Scotland Iron and Steel Inst.*, Vol. XXXVI, Part 6 (March), pp. 83-94.

## Commercial Intelligence

The following are taken from printed reports, but we cannot be responsible for any errors that may occur.

### Mortgages and Charges

[NOTE.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debts due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case, the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.]

**BRITISH AND SAAR STEEL CO., LTD.**, London, S.W.—Registered August 2, by order on terms, £2,200 mortgage, to H. Atkinson, Danecourt, Leicester Road, Bournemouth; charged on Southbourne, Wake Green Road, Birmingham, and land in rear. \*£4,000. July 12, 1929.

**ITABIRA IRON ORE CO. LTD.**, London, E.C.—Registered July 27, £10,000 (not ex.) further charge (supplemental to mortgage dated March 2, 1916, etc.), to Brazilian Investment Syndicate, Ltd., 5, Fenchurch Street, E.C.; charged on properties already charged. \*£641,751. December 31, 1928.

**METAL, ORE AND CHEMICAL CO., LTD.**, London, E.C.—Registered August 8, £5,000 debentures, to R. Pintus, 24, Teignmouth Road, Cricklewood, merchant; general charge. \*Nil. February 2, 1929.

**SHEFFIELD STEEL PRODUCTS, LTD.**—Registered August 15, trust deed dated August 14, 1929, securing £321,366 debenture stock; charged on properties in Sheffield, etc., also general charge. \*£1,687,016. December 11, 1928.

### Satisfaction

**BWANA M'KUBWA COPPER MINING CO., LTD.**—London, E.C.—Satisfaction registered July 23, £4,450, part of amount registered April 13, 1928.

### London Gazette, &c.

#### Companies Winding Up Voluntarily

**ARISTON GOLD MINES, LTD.** By special resolutions August 7, confirmed August 22. S. Hutchinson, Finsbury Pavement House, Moorgate, Chartered Accountant, appointed as liquidator. "That the liquidator be authorised to enter into an arrangement with a new company, to be called Ariston Gold Mines (1929), Ltd., or other suitable name, for the sale to it of the undertaking and assets of this company, subject to its liabilities, in consideration of shares in the new company and the taking over of such liabilities in accordance with the terms of the reconstruction scheme which has been approved by this meeting, and (b) to distribute such shares, or any of them, among the members in accordance with such scheme."

**SOUTH BUKERU (NIGERIA) TIN CO., LTD.** By special resolutions July 29, confirmed August 14. 1. That South Bukeru (Nigeria) Tin Co., Ltd., be voluntarily wound up with a view to its reconstruction, and that H. T. Skipp, 341, Salisbury House, London Wall, Chartered Secretary, be appointed liquidator. 2. That the liquidator be authorised to consent to the registration of a new company to be called "South Bukeru Areas, Ltd." or some other suitable title with a memorandum and articles which have been prepared with the privity and approval of the directors of this company.

# Monthly Metallurgical Section

Published in the first issue of "The Chemical Age" each month.

**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

## Lithium: Its Extraction, Properties and Uses

By G. Malcolm Dyson, Ph.D., A.I.C.

THE recent introduction of lithium alloys into the field of aluminium light alloys has drawn attention to what has hitherto been one of the metallurgically unimportant elements. The element was discovered by Arfvedson, working in the laboratory of Berzelius, and the name "lithium" was given to it (from the Greek, meaning "stony") in order to distinguish it from the alkalis, such as sodium and potassium, which are commonly found in animal and vegetable matter. Lithium is very widely distributed naturally, although in small traces, and in Bunsen's celebrated researches on the application of the spectroscope detailed reference is made to the ease with which the universally distributed traces of lithium are recognised by a persistent line in the dark red. Bunsen, having described his method of detecting lithium, says: "In this way we arrive at the unexpected conclusion that lithium is most widely distributed throughout nature, occurring in almost all bodies. Lithium was easily detected in forty cubic centimetres of water of the Atlantic Ocean . . . in the very pure spring water from the granite in Schleierbach . . . in the ashes of tobacco, in vine leaves, etc."

### Commercial Sources

Various springs contain sufficient lithium to warrant their treatment for its extraction, but this is rather the exception than the general rule, as for example a spring in the Wheal Clifford mine at Cornwall, which contains 372 mg. of lithium per litre. The more usual sources of lithium are the complex silicates, in which part of the more usual alkali metals is replaced by lithium. The more important of these are petalite, a lithium aluminium silicate, and spodumene, a mineral of a similar nature. The list below gives the main minerals containing lithium in workable quantities, together with the more usual amounts to be expected in such minerals:—

MINERAL.	FORMULA.	NATURE.	APPROXIMATE PERCENTAGE OF LITHIUM.
Lepidolite	(see below)	Complex lithium-aluminium silicate	1.3-5.7
(Lithia mica)			
Spodumene	$\text{LiAl}(\text{SiO}_3)_2$	Lithium-aluminium silicate	3.8-5.6
Petalite	$\text{LiAl}(\text{Si}_2\text{O}_5)_2$	Lithium-aluminium silicate	2.7-3.7
Triphylite	$(\text{LiNa})(\text{FeMn})\text{PO}_4$	Complex ferro-manganese phosphate	1.6-3.7

The lithium micas are in reality a complex set of compounds; the lithia mica referred to as lepidolite is held by some mineralogists not to be a true mica, and they suggest that the term "lithium mica" be reserved for the type of mineral found at Killiney Hill, which approximates in formula to  $3\text{FeO} \cdot 2\text{CaO} \cdot \text{Li}_2\text{O} \cdot 4\text{K}_2\text{O} \cdot 4(6\text{Al}_2\text{O}_3 \cdot 16\text{SiO}_2) \cdot 16\text{H}_2\text{O}$ . The distribution of these minerals in workable quantities is comparatively sparse; in this country we have no really large deposits, while there are three deposits in the U.S.A., one at Utö in Sweden, and one each in France and Spain. More recently, the Silver Leaf Mining Syndicate have commenced work upon a lithium deposit in Canada, situated at the Lamprey Falls, Winnipeg River, Manitoba. Here a large number of pockets and lenses of lepidolite and petalite are found disseminated throughout a pegmatite mass 60 ft. wide. The ore is sorted and shipped to this country.

### Extraction

The extraction of lithium takes place by purely chemical treatment of the ores, and the metal itself is obtained by electrolysis of the pure salts. The methods available for the extraction of lithium salts from the ores are divisible into two main groups. Thus from the lepidolite and petalite

group of minerals it is possible to fuse the following mixture, in a finely powdered state, at a temperature of  $1,200^\circ\text{C}$ .: The mixture contains powdered lepidolite together with an equal weight of barium carbonate (the ground mineral witherite can be used here), and half its weight of ground barytes; one-third of the weight of potassium sulphate is also added. During the fusion two separate layers are formed in the melt; a lower layer consisting of barium silicate and sulphate, and an upper layer containing the sulphates of potassium and lithium. The upper layer is easily removed when cold and dissolved in water. The two sulphates are converted into the corresponding chlorides by the addition of the requisite amount of barium chloride, and the liquid, filtered from the precipitated barium sulphate, is evaporated to dryness and extracted with pyridine, in which lithium chloride is soluble and potassium chloride insoluble. This process,

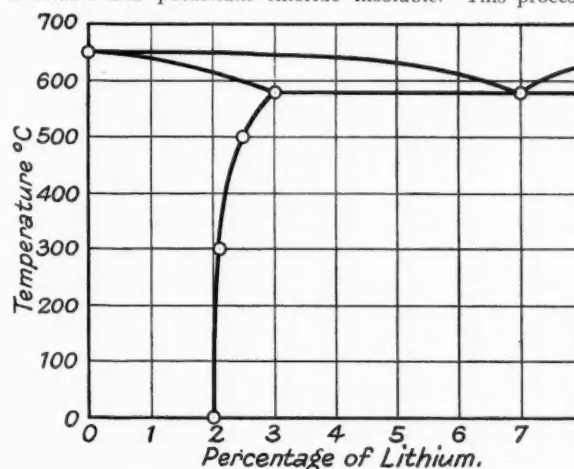


FIG. 1.—THE SYSTEM ALUMINIUM-LITHIUM.

commercially, has several drawbacks; in the first place the high temperature of the fusion is difficult to maintain on an economic scale, and, in addition, the process requires large quantities of expensive barium chloride.

The process has been replaced by a similar one in which the finely powdered ore is heated to a temperature of  $120^\circ\text{C}$ . with concentrated sulphuric acid. The temperature is gradually raised to  $330^\circ\text{C}$ ., at which point the alkali metals are all converted to their respective sulphates. On lixiviating the cooled mass with water, the sulphates of aluminium and lithium dissolve, leaving silica in a state suitable for filtration. The filtered liquor is concentrated, and much of the aluminium sulphate removed by the addition of potassium sulphate, which causes the separation of crystals of potash alum. The residual aluminium is removed by treatment of the concentrated liquor with calcium carbonate and milk of lime. At this point the liquid is further concentrated, and the solution treated with a concentrated solution of potassium carbonate solution, which precipitates the lithium in the form of the carbonate.

When the raw material for the extraction of lithium is the mineral triphylite, a modified form of the Müller process is considered the best. The finely powdered mineral is dissolved in hydrochloric acid, and the iron in the solution oxidised to the ferric state by boiling with nitric acid. Any ferric iron



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### The Production and Uses of Beryllium

By Kurt Illig

(Of the Research Laboratories of the Siemens-Halske A.-G., Berlin)

The following paper was presented to a meeting of the American Electrochemical Society a short time ago. It gives an account of the work carried out at the Beryllium Research Institute, in Germany, and may be regarded as authoritative.

THE year 1928 marked the centenary of the first production of beryllium by the celebrated German investigator, Wohler. During these hundred years almost all of the alkali and alkaline earth metals such as Li, Na, K, Ca, Mg, Ba, and Sr, have been prepared from fused electrolytes in commercial quantities. In the case of beryllium alone did all of the investigators fail, until recently, to prepare this metal in such quantities as to permit a study of its properties and uses. The difficulty which stands in the way of easy isolation of beryllium is its great affinity for oxygen. Before dealing with the modern methods of production, a brief résumé of the history of this interesting metal may be worth while.

#### Occurrence

Beryllium was discovered in 1798 by Vauquelin in the mineral "beryl," which had been known since antiquity. The ordinary beryl, an aluminium-beryllium double silicate, having the empirical formula  $3\text{BeO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$ , has been comparatively unknown; the more valuable varieties of this mineral, the green emerald and the blue-green to pale blue or light rose coloured aquamarine, were known in pre-biblical times.

Other minerals are known to contain more or less beryllium, as for example, chrysoberyl, also named alexandrite ( $\text{AlO}_2)_2\text{Be}$ ; phenacite  $\text{Be}_2\text{SiO}_4$ , which has a rather wide occurrence, and certain rare earth minerals, such as, euclase, gadolinite, helvinite, danalite, bertrandite, leucophane, melinophane—all of which are fundamentally silicates of beryllium, which differ from one another simply by the presence of silicates of other metals. A borate of beryllium, hambergite, and two phosphates, beryllonite and herderite, might also be mentioned.

#### Work of Stock and Goldschmidt

The work which was decisive in the development of beryllium manufacture was accomplished by the German investigators, Stock and Goldschmidt, with the co-operation of Priess and Praetorius. They obtained metallic beryllium for the first time in the form of large reguli, directly from a fused electrolyte.

Investigations have shown that the most desirable conditions are fulfilled in general by the double fluorides of beryllium with sodium and barium. The barium-beryllium fluoride has an appreciable viscosity around  $1,300^\circ\text{C}$ ., but also has the advantage of being only slightly volatile at this temperature range; it has an appreciable dissociation within this range. The sodium-beryllium fluoride is strongly dissociated even at relatively low temperatures. This, therefore, offers a means of obtaining an electrolyte possessing a good conductivity. It has only one disadvantage, namely, that at very high temperatures the separated beryllium metal may be volatilised in appreciable quantities.

If the electrolysis is started with the sodium-beryllium fluoride and with rising temperatures some barium-beryllium fluoride is added in increasing amounts, there is obtained a useful mixture of the double salts for every temperature interval. When the final metal separating temperature of  $1,300\text{--}1,350^\circ\text{C}$ . is reached, it is not difficult to continue the electrolysis for many hours, provided suitable additions of both double salts are made from time to time.

#### The Beryllium Research Institute

Stock and Goldschmidt protected this general method of procedure by letters patent in all large countries. They realised, however, that much additional work was necessary, before all of the details for the continuous production of large quantities of beryllium would be established. In order to study the problems on the broadest basis, the foremost German manufacturers organised a Beryllium Research Institute in 1923, under the chairmanship of the Siemens and Halske A.-G., of Berlin. It was the aim of this institute to make a systematic study of the fundamental Stock-Goldschmidt method in all its details, and to determine whether or not it was possible to produce beryllium metal in commercial quantities from fused electrolytes. The numerous variables which had to be considered made the work tedious, especially so since the temperature range of  $1,300\text{--}1,350^\circ\text{C}$ . had never been used previously in fused electrolytic work. Some of the details of these investigations of the institute to date are herewith recorded.

#### Crucible and Electrode Materials

The question of crucible and of electrode materials had to be settled first. In the early experiments of Stock and Goldschmidt, a crucible made of graphite was used as the anode. Graphite containing as little iron as possible is the only material to use. Acheson graphite is the best. The difficulties encountered in mounting the electrodes of the crucible were soon overcome. Various metals were tried as cathode; the most satisfactory was a special iron, made up into a hollow water-cooled electrode. The electrolysis current is sufficient to maintain the bath at the temperature required. At the start of the process the crucible is filled with pieces of carbon or graphite, the iron carbide is pressed firmly against these pieces, and as soon as the proper temperature is reached the pieces are quickly dumped out of the crucible and a charge of low melting sodium-beryllium fluoride is immediately put in. Then barium-beryllium fluoride is added until the required temperature range is reached for the electrolysis to begin.

Only after a large number of experiments were made was the best method for charging the crucible discovered, which method must be rigidly adhered to. Naturally there will be a relatively large burning loss of the graphite crucible, which, however, may be minimised by surrounding the crucible with another crucible packed with kryptol, to prevent contact between the very hot external wall of the graphite crucible and the air. Another method is to coat the external wall of the graphite crucible with a fused mass of the electrolyte, making an impervious coating, which is in no way objectionable since no impurities will be introduced into the electrolyte.

#### First Production by Above Method

At first, relatively small reguli, of several grams only, could be separated from a single charge, and it was an exciting moment when several reguli of about 20 grams each were obtained. These reguli were still contaminated with the melt, and so were not fit for a scientific or technical investigation. Systematic and progressive development made it possible to obtain a single button of about 50 grams in weight, and at the same time containing only spots of very small amounts of

impurities. It was this material that the several laboratories connected with the Beryllium Institute, as well as others not so connected, investigated. They determined the physical properties of beryllium metal.

Hand in hand with the scientific investigations on fused electrolytes went the determination of authentic methods of analysis. The previous methods recorded in the literature were either too incomplete or entirely too circumstantial to be of undisputed value for rapid work. Very good methods are now available for the rapid determination of beryllium in the presence of aluminium and other metals. In 1926 the Institute felt convinced that in the Stock-Goldschmidt method there was a means by which a commercial production of beryllium metal might be expected. At the same time the commercial applications of this metal were studied.

#### **Commercial Investigations**

On the grounds of the results of the investigations of the Institute during 1926, the Siemens-Halske A.-G. decided to make a systematic study of the applications to a commercial scale. The previous work had shown that the best scientific and technical production of beryllium would be possible only if the salts used were cheaply prepared; especially if they were not hygroscopic, such as the double fluorides. The preparing of the salt from the crude mineral beryl was next investigated. Both of these problems have been solved in the past two years. Simply and cheaply prepared salts, such as the double fluorides, which also give good material yields, are being investigated now.

In the older methods the electrolyte became more and more enriched in barium fluoride, thus shortening the time of a single charge, but with the advent of the new salts it is now possible to make the process continuous and without interruption. Practically speaking, this continuous method is now limited only by the burning of the graphite crucible.

The beryllium metal is in convenient reguli of about 150 grams, varying with the size of the apparatus, and is about 98 per cent. pure. By special methods of remelting the purity can be increased to 99.9 per cent., though the yields of this very pure material are small. The main impurity of our beryllium is iron, which is introduced from the graphite crucible. The recently developed double decomposition methods of the raw beryl for the formation of the electrolyte salts produce about 65 per cent. of beryllium, calculated as metal, so that starting from the mineral the final yield of beryllium metal is about 60 per cent.

#### **Occurrence of Beryl**

While this work was going on, investigations were being made as to the location and size of the beryl deposits in the various parts of the world, and as to the prices at which the material was marketed. This analysis showed that as usual when the possibilities of a new use for a raw material bring it to the attention of the world, it is found in many places. Norway, Spain and especially Florida, Canada and Brazil, in the order of importance, have large deposits of beryl. There appear to be deposits larger than previously realised in south Germany, and in the Ural mountains.

It may be safely predicted that in the near future many beryl deposits will be found because now for the first time mineralogists and geologists have a stimulus for looking for this mineral. The prices for the crude beryl are exceedingly high at present, probably because there is no direct market for the material, but more probably because the owner of a beryl mine has the false impression that he has a mineral classed as a semi-precious stone. The owner must be enlightened as to the actual facts.

#### **Beryllium Alloys**

The most promising field which has so far been recorded in the literature is in the use of beryllium as a light metal. But the first studies of the metallographic division of Siemens-Halske have indicated that such a use is rather remote at present. Pure beryllium is an extremely hard and brittle metal, which scratches glass and can neither be rolled nor drawn because of its coarse crystal structure. Nevertheless plates of about 1 to 2 mm. by 10 to 20 mm. have been made from pure beryllium by special methods for use in X-ray work. But this is no application for which large amounts of metal need be produced.

Since beryllium has a specific gravity of 1.8 it has been considered advisable to try to alloy it with sufficient aluminium to make it workable. In this respect many interesting results may certainly be expected. It was not in the programme of Siemens-Halske to make a systematic investigation in this direction, because the realisation of our ambition to apply beryllium to arts and industry seemed only possible if beryllium alloys were developed which would compete with other alloys.

It has been shown that the addition of 2 to 2.5 per cent. of beryllium to copper or nickel produced alloys possessing properties like those of the bronzes, a fact previously unknown. In the case of additions of beryllium to aluminium the alloy showed properties somewhat like those of duralumin and silumin, though there is no advantage in favour of the beryllium-aluminium alloy. The more expensive beryllium cannot compete with silicon, magnesium, and copper in making alloys with aluminium.

Some important data on beryllium-copper and beryllium-nickel alloys might be noted. Alloys of cobalt with beryllium are very similar, but the silver-beryllium alloys have interesting properties which could not have been determined previously. In the study of beryllium-iron alloys the work has not progressed far enough to permit definite conclusions.

#### **Heat Treatment**

The most outstanding property of the beryllium alloys is in their improvement upon heat treatment. A copper-beryllium alloy containing 2.39 per cent. of beryllium, after ordinary quenching, has a Brinell hardness of 100 kg./sq. mm., but after heat treatment for 17 hours at 300° C. this rises to 440 kg./sq. mm. that is, to over 400 per cent. Some copper alloys containing over 6 per cent. beryllium have a hardness of over 730 Brinell numbers. These alloys cannot be worked in the untreated condition. Bronze-like properties are developed in these copper alloys, which, in the untreated condition, can be rolled, drawn or worked to the desired shape and then by heat treatment can be hardened and roughened. It is possible to make thin elastic pieces. The following information on the strength of these alloys may be of interest.

In the untreated quenched state a copper alloy containing 2.5 per cent. of beryllium has a tensile strength of 48.9 kg./sq. mm., but after heat treatment it is 135 kg./sq. mm. The differences in the transverse bending strength are most astonishing; in the untreated condition, but quenched, it is 60.7 kg./sq. mm., but after heat treatment it rises to 216.7 kg./sq. mm. In the same alloys the yield point changes from 15.7 to 128 kg./sq. mm. on heat treatment, while the elastic limit in the first case is 5 kg./sq. mm., and in the second it is 45.9 kg./sq. mm.

As the beryllium content of these copper alloys is increased the final values for these mechanical properties in the treated conditions are also increased. There is, however, a limit which is 6 to 7 per cent. Be in copper and nickel alloys. In general it may be said that the quenched alloys are very elastic and strong.

Heat treatment increases the elastic limit, and the yield point by about 800 per cent., while the tensile strength and the transverse bending strength increase about 3 to 4 fold. The ductility decreases proportionally with strength. This is not detrimental, since the shaping and working after quenching is done before it is given the heat treatment, so that ductility requirements need not be considered after the treatment. These alloys may be successfully rolled, drawn, and pressed.

#### **Electrical Conductivity**

It is worth noting that the electrical conductivity of these alloys is lowered rather appreciably by the addition of beryllium. Alloys of 2.5 per cent. of beryllium, after quenching at 800° C., have an electric conductivity of 9.8 recip. ohms/sq. mm., of cross section. Heat treatment changes this conductivity to 19 recip. ohms/sq. mm., and after annealing followed by a second hardening it reaches 21.5 recip. ohms/sq. mm. These alloys are more resistant to corrosion than the ordinary bronzes.

Beryllium has an interesting application as a deoxidiser in copper casting. It has been shown that the addition of 0.02 to 0.05 per cent. of beryllium to copper for casting changes the electrical conductivity of the casting from 35 to 50 recip. ohms/sq. mm. The various alloys and their applications have been protected by letters patent.

## Hardness Testing

### The Constant Size Impression Method

Of the various machines developed for the testing of hardness by the indentation method, the Brinell machine is perhaps the most popular and the most widely used, for it allows of the test being quickly and conveniently carried out, and the values obtained can be related in some measure to tensile strength. Various forms of the machine are in use, and it is customary to use hardened steel balls of 1 to 10 mm. diameter as indenter elements, machines using the 1 mm. ball having been designed for testing the hardness of small specimens and thin material. One of the chief limitations of this method of hardness testing arises from the deformation of the ball when testing hard materials.

#### The Rockwell Machine

For measuring the hardness of hardened steels and heat-treated tool steels, the Rockwell machine, where a constant load is used and the depth of penetration measured, is now quite commonly employed. With the development of extremely hard materials for use in the form of tools and dies (such as the tungsten carbide alloys, for example), the need for a satisfactory method of effecting hardness measurements is becoming more necessary. The difficulty of deformation of the indenting tool when measuring high hardness values is overcome by the use of diamond indenters in place of steel, and in various machines this material has been used.

In a recent paper by A. F. Shore read before the American Society for Steel Treating, and printed in *Heat Treating and Forging* for May, 1929, the desirability, in hardness determinations, of reading pressures required to produce a standard impression is dealt with, and the author has designed a machine known as the Monotron hardness indicator in which this principle is employed. This principle overcomes the objection which is sometimes raised against the constant load method, that with soft metals the degree of penetration is relatively large as compared with harder metals, that is, the amount of deformation which the soft metal undergoes is greater and the result is thereby affected.

#### The Monotron Method

Even with the constant size impression method the degree of penetration adopted is of importance, for it is possible that metals having similar elastic limit hardnesses will give very different results with greater penetration if they behave differently under extreme cold working. The errors in measuring high hardness values due to deformation of the steel ball, as in the Brinell machine, are overcome to some extent by the use of a diamond pointed indenter in the Monotron machine.

With a machine embodying the constant size and varying pressure principle, one of the chief difficulties in the way of obtaining accurate measurements of the depth of penetration is due to the elastic shortening of the penetrator element when under load. It is not practicable to make allowances for this factor for different hardness values and in the Monotron machine the depth micrometer is held in a compensator, and so given a retractile movement equal to the elastic shortening of the diamond point when under pressure. The load required to produce a standard penetration, and the depth of the penetration, are read off on two dials which form part of the instrument. For universal application a 0.75 mm. diamond ball point is used, the standard depth of penetration being 0.0018 in., which is 6 per cent. of the ball diameter. With this penetrator the entire hardness range encountered in metals from lead to tungsten carbide alloys can be satisfactorily measured.

#### Method of Making the Test

No special difficulty attaches to the carrying out of the test, irregular shaped pieces of metal being satisfactorily accommodated by a tilting table which can be replaced by a V-block for cylindrical specimens. The values read off on the dial recording pressure are in kilograms and pounds, but other scales can be substituted such as are giving diamond ball Brinell values direct. The modulus of elasticity of the material under test does not affect the result since the depth of the penetration, or hardness value, is read while the load is applied. A protecting cap for the diamond point acts as

a contact for operating the depth gauge, and since the latter moves relative to the position of the cap it has the advantage that any yielding (say of a hollow piece under test) does not affect the result. The depth scale is divided into nine divisions each representing 0.0002 in. or 0.005 mm.

#### Determinations on Very Hard Products

As already indicated, the development of extremely hard metallurgical products has led to increasingly exacting demands being made on hardness testing machines. Of these, surfaces hardened by the nitriding process and tungsten carbide alloys may be cited, the hardness of some of the alloys in this class being twice as much as that of quenched high-carbon steels. Such materials are well beyond the range of hardness which can be satisfactorily determined by hardened steel balls or indenters, and only carefully selected diamonds can be used. Even these must be used with caution on the harder grades of tungsten carbide alloys. The Monotron instrument with its spherical diamond indenter, which gives a straight increment line, can be used to advantage in determining the hardness of these extremely hard products.

In testing materials of this kind, it is advisable to use a depth of penetration corresponding to three divisions only, which does not impose an unduly severe strain on the diamond and yet is quite satisfactory for the purpose. With hardened steel a penetration equal to three divisions requires a pressure of from 35 to 38 kilograms, which when multiplied by three gives the value needed for the standard penetration of nine divisions or 0.0018 in. With superhard steels this value to yield the standard penetration might be as high as 120. With nitrided steel it is of the order of 117, or 39 kilograms for a penetration equal to three divisions.

Values on tungsten carbide alloys vary considerably, one of the softer but tough grades recording 45 kilograms with a depth of three divisions or 135 when calculated to the standard, a harder and more brittle sample yielding corresponding figures of 65 to 75 and 195 to 225 kilograms. On hardened steel, the diamond will last for thousands of tests taken to the full depth of nine divisions, but is not capable of being used on tungsten carbide to this depth.—M. C.

### Estimating Iron in Chromium Alloys

The use of alloys containing iron and chromium, and often also nickel, is at the present time becoming very widespread in engineering, and therefore the following details (supplied by a correspondent) may be of interest to those engaged in the analysis of these alloys. The usual method of separation of iron from chromium is by means of sodium peroxide, but the author has found that the following method gives quite accurate and reliable results on this class of material. The method is based on the oxidation of the chromium by ammonium persulphate and then precipitation of the iron as usual with ammonia. The estimation is carried out as follows:—

From one to three grams of sample is taken (depending on the amount of iron suspected) and dissolved in dilute sulphuric acid if possible. If, however, the alloy is not completely soluble in sulphuric acid, then hydrochloric acid or aqua regia must be used to dissolve it, and then sulphuric acid must be added and the solution evaporated until white fumes are evolved. The solution is then diluted with hot water and boiled. If any silicon is precipitated at this stage it should be filtered off. The solution is now cooled, diluted to 250 ccs., and a measured quantity taken so as to give approximately 0.1 to 0.2 g. of iron in the estimation (a larger quantity may be difficult to filter). This is then made up to about 150 c.c. bulk, about 2 g. of ammonium persulphate crystals added, and the whole boiled for about a quarter of an hour. All the chromium in the alloy will now be oxidised, and any manganese will be precipitated as manganese dioxide, which should be filtered off. Ammonium chloride and ammonium hydrate are now added to the solution to precipitate the iron, which is filtered off, after boiling, and ignited and weighed as ferric oxide as usual. The percentage of iron is then calculated from the weight of sample taken as usual.

The resulting filtrate may sometimes be used for the estimation of nickel in the alloy by the dimethylglyoxime process, but here the chemist must use his discretion, as this depends on the amount of nickel present in the alloy.

## Metallurgical Topics: Monthly Notes and Comments

### From Our Own Correspondents

#### Some Alloys of Aluminium

WITH regard to an article on "Some Alloys of Aluminium," which appeared in this journal last month, Lightalloys, Ltd., of Alpax Works, St. Leonards Road, Willesden Junction, London, write as follows:—

"We notice that in the second column of the article under the heading 'Nickel in Aluminium Bronzes,' there are a number of rather misleading statements. Firstly, it is stated that the addition of nickel to the aluminium bronzes described leads to the formation of a harder and better alloy. 'Aluminium Bronzes' is a term which is usually applied to copper alloys containing a small percentage of aluminium, as for instance, 92 per cent. copper, 8 per cent. aluminium, and not to the high aluminium alloys as described in the previous part of the article; in fact, the whole of the alloys described in the paper are essentially aluminium alloys, and in no wise can be described as aluminium bronzes when that term is used in the popular sense.

"Further, it is stated that among the aluminium bronzes there are three which stand out above all the others, viz., 'Alpax,' 'Lynite' and 'Y' alloy. These three alloys are essentially high aluminium alloys. 'Alpax' certainly derives its name from that of the inventor, Aladar Pacz, but is a silicon aluminium alloy which has been 'modified' by one of the various patented processes. Ordinary aluminium silicon alloy is not 'Alpax,' this term being a trade name for the modified alloy.

"With regard to the strength values given for 'Alpax,' the figure for the alloy described as 'melting under a flux containing sodium fluoride,' is very much lower than is now obtained in ordinary works practice."

#### The Reply

THE author, to whom the above comments have been submitted, replies as follows:—

"In the first place Messrs. Lightalloys object to the use of the term 'aluminium bronze' for alloys which are not composed of a preponderance of copper. This is purely a matter of taste; the term 'bronze' itself signifies an alloy of tin and copper (with or without other additions), and it so happens that with these two metals the alloys which are of most value are those which contain a high percentage of copper. When, however, the term 'bronze' is transferred to aluminium and copper, it is found that both the alloys containing much copper and those containing little copper are very valuable commercially; the trade custom of calling only those alloys high in copper 'aluminium bronzes' is only an artificial distinction. Your correspondents themselves state that these alloys 'can in no wise be described as aluminium bronzes when that term is used in a popular sense.' Articles written for THE CHEMICAL AGE are definitely 'scientific' in contradistinction to 'popular.'

"With reference to the alloy Alpax, I have re-read the section that I wrote on this alloy and must admit that I have not been as clear as could be desired, although I was quite aware that Alpax is a 'modified' aluminium silicon alloy. I have not, however, stated, as your correspondent implies, that ordinary aluminium silicon alloy is Alpax; in fact, I have stated that Alpax is an aluminium silicon alloy the strength of which can be increased (owing to the diminution of silicon grain size) by fusion under a flux containing sodium fluoride. Surely this does not imply that any aluminium silicon alloy is Alpax.

#### Strength of the Alloys

"Further, concerning the strength of these alloys, I would point out the enormous difference that exists between the tensile strength as shown on a special test piece and that shown on a section cut from a casting. With reference to the alloy under discussion I would like to quote the figures of Corson, the acknowledged international authority on the subject, from his treatise on *Aluminium and Its Alloys*. He gives as the tensile strength 30,000 on the separate test piece, but only 19,800 on cast metal. These figures are from an Alpax containing 13 per cent. of silicon and which has been modified by melting under a fluoride flux. Anderson in the *Metallurgy of Aluminium and Its Alloys* gives similar figures. I do not

doubt that the special alloys of Messrs. Lightalloys, Ltd., will often test at considerably above the figures given above when tested on a test piece; but I can state categorically that alloys cast in our own foundry at Loughborough very seldom exceed this figure until after heat treatment."

#### Lead Smelting

THE first of a series of papers to be issued on "Smelting in the Lead Blast Furnace," by G. L. Oldright and V. Miller, has appeared as the August Serial (No. 2,954) of the Bureau of Mines, Washington. It deals with a method for approximating the form of the lead in slag and other products of the lead blast furnace. In the lead blast furnace, as in the blast furnace smelting the iron ores, coke economy is essential and the operator tries not to use more coke than is necessary to metallise the lead in the charge and to separate it from the slag-forming constituents. Over-reduction is contra-indicated, not on grounds of economy alone, but because it leads to accretions of metallic iron, or Speiss, in the crucible, and seriously hampers working. To help in directing aright the operations involved, the forms in which lead may be present in the slags have been ascertained, more particularly as regards its occurrence as metal, or as sulphide, or as silicates, or mixtures of these. These three forms exhaust those in which lead is found to be present in lead blast furnace slags. By treating lead sulphide with brine containing ferric chloride it was found possible to separate it completely from mixtures, while lead silicate can easily be prepared artificially by means which are described and serve to show the nature of reactions to be avoided in actual smelting operations. Methods of analysis are given, and the paper is one which lead smelters should find both useful and suggestive.

#### The Diffusion of Gases through Metals

IN the July issue of the *Revue de Métallurgie*, V. Lombard gives the results of some interesting experiments he has made on the diffusion of gases through metal plates, used as partitions. The gases used were hydrogen, helium and argon, and the metals were nickel, iron and platinum, from which it may be seen that the research was not of an academic nature, but one from which practical results of use in aeronautical engineering and in electric light bulb technology, may be adduced. He has brought out the following facts: (1) The rate of diffusion of hydrogen through nickel plates (those employed varied from 0.160 to 2 millimetres in thickness) does not, provided the temperature be constant, vary in functions of the time, although the amount passed varies a little, according, probably, to the difference in structure of the nickel. The permeability of nickel by hydrogen is not affected by the presence of moisture, or by that of small amounts of oxygen, such as may occur in the hydrogen of commerce; (2) When an inert gas is present the hydrogen passes through the nickel at a rate proportional to the square root of its own pressure, and the total pressure of the mixture exerts no influence on the result. The permeability of nickel to nitrogen and to argon, although small, is not nil, whereas helium permeates it more rapidly than hydrogen. The results with platinum and with iron plates are to be published later.

#### Cementation as a Corrosion Protective

THE fourth, and final, part of the report of the French Committee on the Corrosion of Light and Ultra-Light Alloys, by J. Cournot and E. Perot, deals with cementation methods of protection. The first section is devoted to the influence of the cementation of aluminium by copper; the second deals with the cementation of aluminium and duralumin by various ferro alloys. The process employed was: (1) Direct, by cementing in an electric furnace with the metal itself, and (2) By cementing in a bath of appropriate salts. In the first case adherence of the coating was difficult to secure; in the second, the presence of saline matters may become injurious to the surface of the coated metal. At the temperature of the experiments it was not found possible to cement aluminium by means of ferro-alloys, such as ferro-chrome, etc. On the other hand, in the case of duralumin, "an entectic is precipitated throughout the mass of the metal," due, the authors think, to the expulsion of the copper in the duralumin, and

its combination with some of the aluminium itself. A final section of the research deals with cadmium plating on aluminium and duralumin, and with chromium plating over the cadmium. Indeed, the metals subject to the experiments appear to have first been copper-plated; then cadmium-plated, and, finally, to have been again plated with the chromium, a method which is said to have given very satisfactory results. Samples so treated bent well, without peeling, and even stoop drawing, without revealing any signs of cracking so far as the surface platings were concerned.

### **Pure Iron**

ALL that is known, and has hitherto been put on record with reference to the physical, thermal, magnetic and electrical properties of pure iron, will be found summarised and compiled from the wide literature of the subject in Bulletin 296 of the Bureau of Mines. This is but a section of the contents of this admirable publication, which is by Oliver C. Ralston, and ostensibly deals only with the subject of its official title "Iron Oxide Reduction Equilibria." Its secondary title amplifies this somewhat, being a "A Critique from the Standpoint of the Phase Rule and Thermodynamics." In point of fact, it contains a good deal more than its title implies and is a most exhaustive summary not only of its subject but of many incidental and accessory aspects of pure iron and iron oxide metallurgy.

The main headings of the sections indicate the scope of the work; they are: Ferric Oxide; Higher Oxides of Iron; The System Ferric-Oxide Magnetite; Magnetite, Iron-Oxygen Intermediate Complexes; Ferrous Oxide; Iron-Oxygen Complexes; The System Iron; and The System Iron-Carbon. The work will be valued, as the matter it contains, conveniently grouped and compiled, would otherwise have to be pursued through countless transactions of innumerable societies and institutions. Yet the data given are precisely those of which the iron metallurgist is most often in need, and usually in doubt as to where to find them.

### **Steel "Failures" and Margins of Safety**

THE failure of the bolts in an aeroplane disaster that cannot as yet be regarded as ancient history has given engineers and metallurgists, alike, furiously to think. That one bolt should fail is regrettable, but, in existing circumstances, not unnatural. That a second should fail is bad. That, with extra duty, one or two more failed likewise, could, probably, be anticipated. The trouble is that it was not anticipated. The margin of safety seemed ample, but certain percentages of failure, or rejection of a given lot of bolts for use in circumstances of intensely severe stress, which may at any time become wholly exceptional, should ensure the rejection of the lot. Alternatively, as a contemporary rightly suggests, a factor of safety might be adopted, based not on strength or service stresses but on time of service or endurance. That is, after a length of service bearing some proportion to the ascertained fatigue strength of the more severely stressed part or parts, the whole system should be overhauled and such parts replaced. This, of course, still leaves open the question of occasional sudden and unexplained failure, of the kind that no preliminary testing or method of inspection can at present guard against.

### **"Natural" Chromium-Nickel Steels**

FOR certain parts of certain machines, expense is no object. The best and strongest material alone should be used; "strong," that is, in respect not of any generalised or theoretical stresses, but in resisting just those special, extraordinary and cumulative stresses the part is called upon to bear in practice. This is, of course, the merest truism. Nevertheless, it is readily conceivable that material better than that habitually employed should, if possible, be used, and the resources of metallurgy are surely equal to making a material that will stand up to anything—for a time—and of replacing before that time has elapsed. A description was given by Mr. Arthur Richards, more than twenty years ago, of an extraordinarily shock-stress-resistant steel made, somewhat fortuitously, at Bolckow Vaughan's works, Middlesbrough. It was made from pig iron that had itself been made from a Greek ore containing notable percentages of chromium and nickel. It was difficult to smelt, and very refractory, and the resulting pig iron contained 1.750 per cent. of nickel and cobalt, 4 per cent. of chromium, and 0.4 per cent. of silicon. It

was blown in an acid Bessemer converter and made an exceptionally tough steel with 1.4 per cent. of nickel, 0.25 per cent. of cobalt, and 0.32 per cent. of chromium. On a Stead-Richards machine such steel withstood 210,910 revolutions to fracture, as against 77,333 for a 0.46 carbon steel, and 100,175 in the case of a silicon steel (carbon, 0.45; silicon, 0.24). Later on the Mayari ores of Cuba, which contain similar constituents, were successfully employed to produce a very similar steel, with rather more chromium, which likewise gave remarkable results. In the case of monel metal, what is called a "natural alloy" has been shown to be superior to one deliberately made to as nearly the same composition as possible. Automobile and aeronautical engineers and metallurgists might be well advised to test the virtues of a "natural steel" alloy of the kind described, and if it be not now procurable there is no reason why it should not be made, seeing that ores which contain the necessary constituents in the necessary quantities are fairly abundant.

### **Steel Strip and Bar**

WE have received from United Strip and Bar Mills, Ltd., Sheffield, a handy pocket-size booklet setting out the various sections of steel strip and bar they have available for their customers' use. Many useful tables are also incorporated. The United Strip and Bar Mills, Ltd. (which is a branch of The United Steel Companies, Ltd.) caters specially for the reinforced concrete trade. The company has a service department whereby reinforcement bar is supplied in lengths, ready bent, hooked, marked and bundled, and delivered to suit the contract, thus relieving the contractor of an arduous duty on site. A special department under a ferro-concrete engineer will also get out quantities and advise generally on ferro-concrete construction for the needs of the smaller contractor. Steel users in the South are catered for by the large stocks for immediate delivery held at the London warehouse. Those interested may obtain a copy of this useful booklet on application to the Publicity Department, United Strip and Bar Mills, Ltd., The Ickles, Sheffield.

### **A New Technical College**

THE Constantine Technical College, Middlesbrough, which the Prince of Wales is to open officially next spring, will be opened for students on September 16, when courses for day and evening students will begin. The college has cost £80,000 to build and £45,000 to equip, and is in the charge of Dr. D. H. Ingall, who anticipates that between 700 and 800 students will be enrolled. Special attention is being paid to the industrial needs of the district, and the college is certain to play an important part in the supply of highly trained technical and semi-technical workers needed in the great industries of Tees-side. The courses include one for iron and steel operatives, which has been designed for those who, while not desiring to take the higher courses in metallurgy, can secure a certain amount of scientific training. The equipment of the college, towards the cost of which substantial donations have been made by the principal firms in the district, includes a high frequency electric melting furnace and the largest experimental rolling mill installed in any technical school in the country.

### **Abnormality in Case-Carburised Steel**

DIFFERENT results on depth of carbon penetration, grain size and uniformity of surface hardness are often found in carburised steels of essentially the same chemical analysis, and the term "abnormality of case-carburised steel" has been used to denote certain of these variations.

A study of this perplexing problem is being conducted at the Pittsburg Experiment Station of the United States Bureau of Mines, Department of Commerce. It has been found that steels may show two distinct types of abnormality on carburisation; grain size abnormality and structural abnormality. The first is usually due to the addition of certain elements to the steel and may be regulated by the elements added in the manufacture of the steel. Structural abnormality has been found to be associated with either a high FeO content or with the presence of very small inclusions in the experimental steels made by the Bureau of Mines. Apparently as the inclusions become larger they affect structural abnormality less and less. Heat treatments do not affect this type of abnormality.

## Trade, Commerce, Finance : The Month in Review

### From Our Market Correspondent

AUGUST has maintained its reputation as the holiday month, and the iron and steel trade during that period has been particularly quiet. The annual closing down of works for holidays or repairs or both has been general throughout the country, and both production and consumption have been materially curtailed. The reduction in the volume of business has been gradual over several weeks, and as the mills were executing orders more quickly than new ones were coming in, the stoppage of ten days or a fortnight gave a welcome opportunity of accumulating orders so as to give a good start when the autumn revival in trade sets in.

#### Improving Prospects

There is nothing abnormal in this position. It is the usual experience year after year, and the steel makers are prepared for it. With the advent of September a change for the better may be expected, and already there are signs that business is becoming more brisk. The general expectation is that the last quarter of the year will prove to be quite good for the steel works. Naturally during the past few weeks there has been very little buying, as most consumers had covered themselves for their requirements for a month or two ahead and the holiday season does not stimulate fresh buying, but the feeling is that by the middle of September there will be a considerable amount of new business transacted.

It is too much to expect that all the steel works will be fully employed during the closing months of the year. That is a condition which may be hoped for but which is extremely unlikely to be attained in the present stage of development of the iron and steel industry. So far, there have been no really effective steps taken to bring productive capacity into economic relation with the demand, and until that is done there is no alternative but to have a number of plants working part time. That is the prospect for the great majority of the works, and it is the problem which will have to be solved before prosperity can return to the industry as a whole. The steel makers are fully alive to this fact, and in some of the recent amalgamations action is already being taken along this line.

#### Rationalisation of Orders

Where within the control of one group there are several plants capable of turning out the same class of work, it is far better that the available orders should be so distributed that some of these plants are working full time, even if others are closed down altogether, rather than having all of them working intermittently. This policy is bound to involve hardship not only on the workmen but on the staff; yet it is the only way to stop or at least to minimise the ridiculous cutting of prices which is being carried on by some of the works who in their anxiety to divert orders to themselves appear to have lost their sense of business values. Special qualities of steel which rightly command an extra price and for which the customer has always been willing to pay this extra are being offered at practically the same price as ordinary steel; boiler plates are being sold at very little more than ship plates; and so the ground is being cut from under the feet of those makers who have devoted their energies to the production of the highest quality of steel.

It is always easy to get orders by reducing prices, but it is extremely difficult to restore prices to their former level. Definite instances could be given of reductions of 10s., 20s. and even 25s. per ton in the prices of special steels, with the sole object of filling the order book, with the result that what had previously been a legitimate profit to one works disappears or is turned into a loss to the other makers who have needlessly cut the price. As already stated, this kind of thing will only cease when the offenders have a sufficiently good order book to remove the necessity of poaching on the grounds of their competitors.

#### Amalgamations

The latest amalgamation in the steel trade is that of Dorman, Long and Co. and Bolckow Vaughan and Co. on the North East coast. Rumours of it had been prevalent for some time. The amalgamation is a natural one. The undertakings are situated fairly close together and their activities are similar in many respects. The fusion should offer good scope for the avoidance of overlapping, the cutting out of superfluous plant

so as to ensure the most favourable concentration of production, and the simplification of administration. This process of amalgamation or absorption is going steadily on.

Another old established steel works, with one of the most honoured names in the history of the industry, is about to disappear. The works of Henry Bessemer and Co., Ltd., of Sheffield, have been taken over by another local firm of wheel and axle makers, and they will shortly be closed down for good. After a long and successful career they, like so many others, fell on evil times, and for the past few years have been struggling to keep going. Now their end has come. It is all part of the process of reorganisation of the steel industry which can be no longer delayed.

#### Expenditure on Plant

Another sign of this reorganisation is the expenditure on new plant which is being incurred by many of the works. Considerable sums are being expended with the object of installing the latest methods of production so as to reduce the cost. Selling prices move upwards very slowly, nothing like fast enough to keep pace with the advance in prices of raw materials, and the steel makers are again forced to concentrate on the reduction of manufacturing costs so as to maintain some margin between cost and selling price. The time and skill which have been devoted to the improvement of steel works practice have opened up a field of possibilities for the steel maker, and there ought to be no hesitation in taking advantage of them when once the difficulty of finding the necessary capital has been overcome.

#### General Market Conditions

There is not much to be said about general market conditions during the past month. The quietness has been common to all sections, although the pig iron market has been the least affected. The output from the blast furnaces has been fully absorbed, and still the demand has not been completely satisfied. The production of iron during the stoppages of the steel works has helped in overtaking arrears. There has been no sign of any weakening in prices. The recent advances have been firmly maintained, and a further upward move is not unlikely as soon as the works get into full swing again.

The blast furnace owners are expecting a good demand for iron on forward contracts and they will probably seek to cover themselves against probable increase in cost. The price of blast furnace coke is still steadily moving upwards, and this movement is not likely to be checked by the increase in output which it is hoped to achieve. The hematite makers also are predicting higher prices in the near future, as the present prices show no margin of profit. It is not possible to forecast any move in steel prices.

The official prices for the controlled products remain unchanged, while for the uncontrolled steels, such as boiler plates and small bars, there has been a slight weakening. Boiler plates have been sacrificed in order to secure contracts for tank plates, and small bars are meeting with more severe competition from the Continent. Nevertheless, the steel makers are likely to be faced with increased costs in fuel and scrap and it would therefore be only reasonable if selling prices were suitably adjusted. It would not be surprising if the associated steel makers decided to ask a further advance for steel plates and sections, but the competition for the uncontrolled materials is so keen that advances can hardly be expected.

The output of pig iron for July was 671,900 tons compared with 657,800 tons in June, there being two more furnaces in blast. The output of steel was 804,800 tons compared with 830,900 tons in June.

#### "Aluminium Facts and Figures"

A FURTHER series of data sheets for inclusion in their handbook, "Aluminium Facts and Figures," has been issued by the British Aluminium Co. A.73 and A.74 show enlarged tables of the properties of stranded aluminium and steel-cored aluminium conductors, revised in conjunction with the British Engineering Standards Association. A.166 is a new data sheet on expanded aluminium sheets. The metal in this form makes up into economical framing, backgrounds and screening, and a variety of light structural uses. Further data will be issued from time to time.

## Some Inventions of the Month

By Our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature, published weekly in THE CHEMICAL AGE.

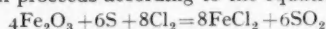
### Alloys

AN alloy suitable for electric cable sheathings and other articles comprises lead with 0.02-0.1 per cent. of calcium, the product being heated to 310-325° C., quenched, and aged. The article may be made from the alloy either before or after the heating. See Patent Application 314,522, bearing the International Convention date June 29, 1928, by Standard Telephones and Cables, Ltd., of London, Assignees of R. S. Dean, of Riverside, Illinois, U.S.A.

### Extracting Metals

ZINC, iron, and other metals are extracted from their ores by treatment with acid on the countercurrent principle, purification of the resulting solutions by means of the sulphides of the respective metals, and recovery of the metals by electrolysis. The purification is preferably effected by aspirating the solution through a cake of the sulphide. A process and an apparatus for the extraction of zinc by sulphuric acid from roasted or oxidic ores, with secondary recovery of copper, cadmium, lead, silver, arsenic, antimony, nickel, and cobalt, are described and illustrated in detail. See Patent Application 313,858, bearing the International Convention date June 15, 1928, by L. Sturbelle, of Brussels.

IN a process for the extraction of iron from oxide ores by chlorination, described in Specification 316,404, dated July 3, 1928, by W. S. Millar, of London, the ore is mixed with sulphur or sulphide ore or material such as pyrites, chalcopyrite, pyrrhotite, mattes, spent oxide, etc., and the mixture is heated with chlorine or a gas containing free or combined chlorine, such as sulphur chlorides or volatilised ferric chloride. In the simplest case, using sulphur and chlorine, the reaction proceeds according to the equation



Non-ferrous metals present are also converted into chlorides, and the chlorine may be recovered from the various metallic chlorides by electrolysis or oxidation and used for the treatment of a further quantity of the ore-sulphur or ore-sulphide mixture. Two methods of separation of the non-ferrous metals from the iron are described in detail, one being based on the separation of ferrous chloride by crystallisation from a concentrated aqueous solution of the product from the chlorination, and the other on the separation by volatilisation of ferric chloride formed by subjecting the product from the chlorination to the oxidising action of chlorine or of limited quantities of air or oxygen at a moderate temperature. The chlorination reaction is strongly exothermic and the desired temperature is maintained by regulating the rates of admission of the reagents.

### Iron

A PROCESS and apparatus for reducing iron ores and utilising the hot products of the reduction for cracking oils are described and illustrated in detail in Patent Application 313,597, bearing the International Convention date June 16, 1928, by Trent Process Corporation of New York, U.S.A. A mixture of ore and carbonaceous material is heated in a furnace, and the iron produced is deposited in oil which thus protects it from oxidation, while the oil vapours rising in the furnace are cracked, by contact with the hot reduced ore, and the products of the cracking are removed. The ore may in some cases be used without the carbonaceous material, the reduction being effected by the oil vapours.

### Iron Alloys

RUSTLESS or stainless iron alloys of a carbon content not exceeding 0.1 per cent. are obtained by a process described in Specification 315,444, dated February 14, 1928, by L. F. Reinartz and J. H. Nead, of Middletown, Ohio, U.S.A. A bath of molten iron, of very low carbon content and very low impurity range under oxidising conditions, is prepared in an electric furnace, preferably by diversion of molten ingot iron from an open hearth furnace. The molten iron is then degasified in the electric furnace, and a reducing slag is formed on the surface of the "killed" iron, whereupon an alloying metal, substantially free from carbon, is added while maintaining the temperature of the furnace. Specified alloying metals are chromium, nickel, vanadium, cobalt, molybdenum, and titanium.

ALLOYS of iron with chromium, manganese, tungsten, or the like, having a ratio of alloying metal to carbon below 30, are decarburised with little or no external heating by subjecting them to a blast enriched to contain not less than 48 per cent. of oxygen so that a temperature of at least 1,600° C. is maintained by oxidation of the constituents of the alloy, the blast being applied to the surface of the molten alloy so as to protect the linings of the containing vessel from the intense heat, and the blowing being continued until the ratio of alloying metal to carbon is above 30. The final decarburisation to the desired limit may be effected by blowing with hydrogen to effect the elimination of the carbon with relatively small loss of alloying metal. The hydrogen is preferably introduced beneath the surface of the molten metal. See Spec. 316,329 (Potts), dated March 27, 1928, a communication from Electro Metallurgical Co., New York.

### Platinum

A PROCESS for the recovery of metals of the platinum group from ores, concentrates, mattes, or residues containing them, especially ores in which they are associated with sulphides of nickel, copper, or iron, is described in Specification 316,063, dated May 1, 1928, by A. R. Powell, of Amersham, Bucks, E. C. Deering, of Barnet, Herts, and Johnson Matthey and Co., Ltd., of London. A matte containing the precious metals is first formed by smelting the ore, etc., and this matte is treated to form or provide within it a proportion of free metal of the iron group, especially iron and nickel, which acts as a collector for the precious metals. A disintegrating agent, such as an alkali hydroxide, carbonate, or sulphide, is added to the molten matte, from which, on cooling, the metal crystallises out. The cold matte is crushed and the meta is separated from the sulphides by magnetic or mechanical means.

### Separating Ores

A PROCESS for facilitating the recovery of oxidised ores, especially of copper, in their separation by froth flotation methods is described in Specification 314,822, dated March 3, 1928, by R. J. Lemmon, of London. The ore particles are treated, during or prior to the froth flotation, with a small proportion of a thiocyanogen compound. Specified compounds are esters, salts, or other derivatives of isothiocyanic acid, decomposition products formed by treating a cyanogen sulphide, or an isomer or alkyl derivative thereof, with alkalies and isopersulphocyanic or isodithiocyanic acid or their soluble salts or esters. Such compounds may be used either alone or in admixture with additional sulphur.

### Treatment of Ores

OXIDE ores, particularly of tin, and superficially oxidised sulphide ores, are subjected to a superficial reduction preliminary to separation by flotation. The reducing agent may be used in solution, or hydrogen or a gas containing it may be added or generated in the flotation apparatus. See Patent Application 311,239, bearing the International Convention date May 7, 1928, by J. Barnitzke, of Clausthal, Harz, Germany.

A METHOD of extracting zinc and like volatile metals from oxidised ores is described in Patent Application 312,667, bearing the International Convention date May 30, 1928, by A. Folliet and N. Sainderichin, of Paris. A current of air, heated to 650 to 800° C., is blown over a charge comprising a mixture of fine ore with coal or lignite and a small quantity of an alkali chloride, e.g., in the form of sea water. In the preferred arrangement the air is delivered through a tangential nozzle at the lower end of an inclined rotary furnace having communicating compartments, and the volatile metal is discharged at the top to a condenser, filter, etc.

ORES or metallurgical products containing metals capable of conversion into carbonyls are rendered amenable to the action of carbon monoxide to effect such conversion by removing or rendering innocuous any easily fusible or slag-forming substances such as zinc, cadmium, tin, bismuth, lead, or silicic acid combined with alkalies, lead, etc. Zinc or cadmium may be removed by volatilisation in an inert or reducing atmosphere. A material containing iron and zinc may be oxidised and thereafter treated with hydrogen at 475° C. to reduce the iron, but not the zinc. Slag-forming substances may be removed by flotation or by magnetic separation. See Patent Application 312,629, bearing the International Convention date May 29, 1928, by I.G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany.

## Current Articles Worth Noting

*We give below a brief index to current articles in the technical Press dealing with metallurgical subjects.*

**ALLOYS.**—The structure of gold-nickel alloys. W. Heike and H. Kessner. *Zeitschrift anorganische Chem.*, Vol. 182, Part 3, pp. 272-280 (in German).

Investigations on the stability of the hysteresis of iron-nickel alloys. G. Gossels. *Zeitschrift anorganische Chem.*, Vol. 182, Part 3, pp. 19-27 (in German).

Germanium. XXXI. Alloys of germanium: silver-germanium. T. R. Briggs, R. O. McDuffie and L. H. Willisford. *Journal Physical Chem.*, July, pp. 1080-1096.

**COPPER.**—The recrystallisation of electrolytic copper after hot rolling. A. Tafel. *Zeitschrift Metallkunde*, August, pp. 265-267 (in German).

**CORROSION.**—The effect of various salts on the dissolution of pure aluminium in hydrochloric acid. J. Calvet. *Comptes Rendus*, July 22, pp. 183-188 (in French).

The corrosiveness of soils with respect to iron and steel. H. D. Holler. *Industrial and Engineering Chem.*, August, pp. 750-755. A method of studying soil corrosion based on the relation of salt content, acidity, and corrosiveness of soil to rainfall is suggested. The corrosiveness of soils as indicated by the initial losses of buried specimens in humid areas of the United States has been correlated with their acidity.

The artificial corrosion of Japanese special alloys. D. Uno. *Korrosion und Metallschutz*, June, pp. 122-130; July, pp. 147-156 (in German).

**ELECTRODEPOSITION.**—Electrolytic deposits on aluminium and its alloys. H. K. Work. *Revue de Métallurgie*, July pp. 378-390 (in French).

**ELECTROMETALLURGY.**—The electrolytic production of metals: To-day's methods and their practical application. G. Eger. *Chemische Fabrik*, July 10, pp. 323-324; July 17, pp. 333-335; July 31, pp. 351-352 (in German). Deals with the production of copper, the noble metals, zinc, cadmium, lead, tin, nickel, cobalt and iron; and also with chromium and cadmium plating.

The separation of bismuth by rapid electrolysis in acid liquor. A. Jilek and J. Lukas. *Collection Czechoslovak Chemical Communications*, July, pp. 369-376 (in French).

Advances in the domain of zinc electrolysis with especial regard to the Tainton process. G. Eger. *Metall und Erz*, Vol. XXVI, Part 15, pp. 373-383 (in German). The development of zinc sulphate electrolysis according to the processes used hitherto; the more important fundamentals of the process, especially the effects of changing acid content and changing current density; the chief characteristics of the Tainton process—strongly acid solutions in lixiviating and high current density; an account of the great installation at Kellogg, Idaho.

**FATIGUE.**—High frequency fatigue. C. F. Jenkin and G. D. Lehmann. *Proc. Roy. Soc. A*, August, pp. 83-119. The object of the researches described was to determine the effect of frequency of alternation of stress on the fatigue limits of various metals. Tests at frequencies up to 20,000 periods per second were made on rolled, normalised and hardened steel; rolled aluminium; annealed copper and normalised Armco iron.

**GENERAL.**—The content of hydrogen and carbon monoxide of some metals melted *in vacuo*. A. Villachon and G. Chaudron. *Comptes Rendus*, August 12, pp. 324-326 (in French).

The colouring of metals. I. and II. H. Kurrein. *Chemiker-Zeitung*, August 7, pp. 609-610; August 14, pp. 630-631 (in German).

Bimetals. W. Rohn. *Zeitschrift Metallkunde*, August, pp. 259-264 (in German). Bimetals—i.e., strips formed of two metallic layers of different coefficient of expansion.

The solubility of gases in melts of pure aluminium and of an aluminium alloy. W. Claus, with S. Briese-meister and E. Kalaehne. *Zeitschrift Metallkunde*, August, pp. 267-268 (in German). Investigations on the solubility, in melts of pure aluminium and an American alloy (8 per cent. copper and 92 per cent. aluminium), of nitrogen, oxygen, carbon monoxide and dioxide, sulphur dioxide, hydrogen, illuminating gas, methane, ethylene and water vapour; a note on Y alloy.

Investigations on the diffusion of gases through metals. V. Lombard. *Revue de Métallurgie*, July, pp. 343-350 (in French). Deals with hydrogen-nickel; iron, platinum-hydrogen; and nickel-nitrogen, argon, helium.

A high-temperature modification of manganese. E. Persson and E. Ohman. *Nature*, August 31, pp. 333-334.

**IRON.**—Investigations of equilibria in the reduction, oxidation, and carburisation of iron. VIII. R. Schenck, T. Dingmann, P. H. Kirscht und H. Wesselkock. *Zeitschrift anorganische Chem.*, Vol. 182, Part 3, pp. 97-117 (in German). The system iron-oxygen.

**STEEL.**—Elastic limit steel. F. G. Martin. *Journal West of Scotland Iron and Steel Inst.*, Vol. XXXVI, Part 6 (March), pp. 83-94.

## Commercial Intelligence

*The following are taken from printed reports, but we cannot be responsible for any errors that may occur.*

### Mortgages and Charges

[NOTE.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debts due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case, the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.]

**BRITISH AND SAAR STEEL CO., LTD.**, London, S.W.—Registered August 2, by order on terms, £2,200 mortgage, to H. Atkinson, Danecourt, Leicester Road, Bournemouth; charged on Southbourne, Wake Green Road, Birmingham, and land in rear. \*£4,000. July 12, 1929.

**ITABIRA IRON ORE CO. LTD.**, London, E.C.—Registered July 27, £10,000 (not ex.) further charge (supplemental to mortgage dated March 2, 1916, etc.), to Brazilian Investment Syndicate, Ltd., 5, Fenchurch Street, E.C.; charged on properties already charged. \*£641,751. December 31, 1928.

**METAL, ORE AND CHEMICAL CO., LTD.**, London, E.C.—Registered August 8, £5,000 debentures, to R. Pintus, 24, Teignmouth Road, Cricklewood, merchant; general charge. \*Nil. February 2, 1929.

**SHEFFIELD STEEL PRODUCTS, LTD.**—Registered August 15, trust deed dated August 14, 1929, securing £321,366 debenture stock; charged on properties in Sheffield, etc., also general charge. \*£1,687,016. December 11, 1928.

### Satisfaction

**BWANA M'KUBWA COPPER MINING CO., LTD.**—London, E.C.—Satisfaction registered July 23, £4,450, part of amount registered April 13, 1928.

### London Gazette, &c.

#### Companies Winding Up Voluntarily

**ARISTON GOLD MINES, LTD.** By special resolutions August 7, confirmed August 22. S. Hutchinson, Finsbury Pavement House, Moorgate, Chartered Accountant, appointed as liquidator. "That the liquidator be authorised to enter into an arrangement with a new company, to be called Ariston Gold Mines (1929), Ltd., or other suitable name, for the sale to it of the undertaking and assets of this company, subject to its liabilities, in consideration of shares in the new company and the taking over of such liabilities in accordance with the terms of the reconstruction scheme which has been approved by this meeting, and (b) to distribute such shares, or any of them, among the members in accordance with such scheme."

**SOUTH BUKERU (NIGERIA) TIN CO., LTD.** By special resolutions July 29, confirmed August 14. 1. That South Bukeru (Nigeria) Tin Co., Ltd., be voluntarily wound up with a view to its reconstruction, and that H. T. Skipp, 341, Salisbury House, London Wall, Chartered Secretary, be appointed liquidator. 2. That the liquidator be authorised to consent to the registration of a new company to be called "South Bukeru Areas, Ltd." or some other suitable title with a memorandum and articles which have been prepared with the privacy and approval of the directors of this company.





# Monthly Metallurgical Section

Published in the first issue of "The Chemical Age" each month.

**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

## Lithium: Its Extraction, Properties and Uses

By G. Malcolm Dyson, Ph.D., A.I.C.

THE recent introduction of lithium alloys into the field of aluminium light alloys has drawn attention to what has hitherto been one of the metallurgically unimportant elements. The element was discovered by Arfvedson, working in the laboratory of Berzelius, and the name "lithium" was given to it (from the Greek, meaning "stony") in order to distinguish it from the alkalis, such as sodium and potassium, which are commonly found in animal and vegetable matter. Lithium is very widely distributed naturally, although in small traces, and in Bunsen's celebrated researches on the application of the spectroscope detailed reference is made to the ease with which the universally distributed traces of lithium are recognised by a persistent line in the dark red. Bunsen, having described his method of detecting lithium, says: "In this way we arrive at the unexpected conclusion that lithium is most widely distributed throughout nature, occurring in almost all bodies. Lithium was easily detected in forty cubic centimetres of water of the Atlantic Ocean . . . in the very pure spring water from the granite in Schleierbach . . . in the ashes of tobacco, in vine leaves, etc., etc."

### Commercial Sources

Various springs contain sufficient lithium to warrant their treatment for its extraction, but this is rather the exception than the general rule, as for example a spring in the Wheal Clifford mine at Cornwall, which contains 372 mg. of lithium per litre. The more usual sources of lithium are the complex silicates, in which part of the more usual alkali metals is replaced by lithium. The more important of these are petalite, a lithium aluminium silicate, and spodumene, a mineral of a similar nature. The list below gives the main minerals containing lithium in workable quantities, together with the more usual amounts to be expected in such minerals:—

MINERAL.	FORMULA.	NATURE.	APPROXIMATE PERCENTAGE OF LITHIUM.
Lepidolite (Lithia mica)	(see below) ..	Complex lithium-aluminium silicate	1.3-5.7
Spodumene ..	$\text{LiAl}(\text{SiO}_3)_2$	Lithium-aluminium silicate .....	3.8-5.6
Petalite .....	$\text{LiAl}(\text{Si}_2\text{O}_5)_2$	Lithium-aluminium silicate .....	2.7-3.7
Triphylite ....	$(\text{LiNa})(\text{FeMn})\text{PO}_4$	Complex ferro-manganese phosphate	1.6-3.7

The lithium micas are in reality a complex set of compounds; the lithia mica referred to as lepidolite is held by some mineralogists not to be a true mica, and they suggest that the term "lithium mica" be reserved for the type of mineral found at Killiney Hill, which approximates in formula to  $3\text{FeO} \cdot 2\text{CaO} \cdot \text{Li}_2\text{O} \cdot 4\text{K}_2\text{O} \cdot 4(6\text{Al}_2\text{O}_3 \cdot 16\text{SiO}_2) \cdot 16\text{H}_2\text{O}$ . The distribution of these minerals in workable quantities is comparatively sparse; in this country we have no really large deposits, while there are three deposits in the U.S.A., one at Utö in Sweden, and one each in France and Spain. More recently, the Silver Leaf Mining Syndicate have commenced work upon a lithium deposit in Canada, situated at the Lamprey Falls, Winnipeg River, Manitoba. Here a large number of pockets and lenses of lepidolite and petalite are found disseminated throughout a pegmatite mass 60 ft. wide. The ore is sorted and shipped to this country.

### Extraction

The extraction of lithium takes place by purely chemical treatment of the ores, and the metal itself is obtained by electrolysis of the pure salts. The methods available for the extraction of lithium salts from the ores are divisible into two main groups. Thus from the lepidolite and petalite

group of minerals it is possible to fuse the following mixture, in a finely powdered state, at a temperature of  $1,200^\circ\text{C}$ .: The mixture contains powdered lepidolite together with an equal weight of barium carbonate (the ground mineral witherite can be used here), and half its weight of ground barytes; one-third of the weight of potassium sulphate is also added. During the fusion two separate layers are formed in the melt; a lower layer consisting of barium silicate and sulphate, and an upper layer containing the sulphates of potassium and lithium. The upper layer is easily removed when cold and dissolved in water. The two sulphates are converted into the corresponding chlorides by the addition of the requisite amount of barium chloride, and the liquid, filtered from the precipitated barium sulphate, is evaporated to dryness and extracted with pyridine, in which lithium chloride is soluble and potassium chloride insoluble. This process,

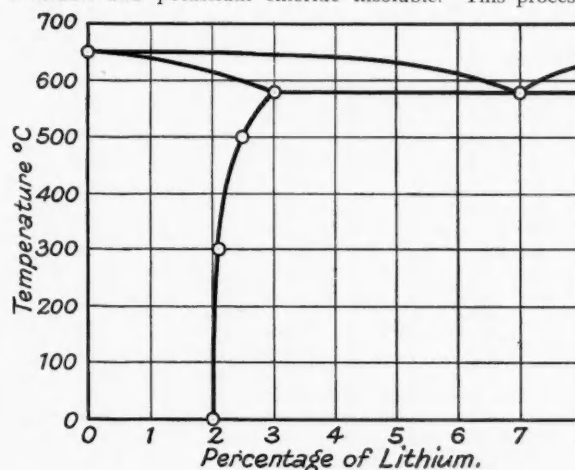


FIG. 1.—THE SYSTEM ALUMINIUM-LITHIUM.

commercially, has several drawbacks; in the first place the high temperature of the fusion is difficult to maintain on an economic scale, and, in addition, the process requires large quantities of expensive barium chloride.

The process has been replaced by a similar one in which the finely powdered ore is heated to a temperature of  $120^\circ\text{C}$ . with concentrated sulphuric acid. The temperature is gradually raised to  $330^\circ\text{C}$ ., at which point the alkali metals are all converted to their respective sulphates. On lixiviating the cooled mass with water, the sulphates of aluminium and lithium dissolve, leaving silica in a state suitable for filtration. The filtered liquor is concentrated, and much of the aluminium sulphate removed by the addition of potassium sulphate, which causes the separation of crystals of potash alum. The residual aluminium is removed by treatment of the concentrated liquor with calcium carbonate and milk of lime. At this point the liquid is further concentrated, and the solution treated with a concentrated solution of potassium carbonate solution, which precipitates the lithium in the form of the carbonate.

When the raw material for the extraction of lithium is the mineral triphylite, a modified form of the Müller process is considered the best. The finely powdered mineral is dissolved in hydrochloric acid, and the iron in the solution oxidised to the ferric state by boiling with nitric acid. Any ferric iron

in solution is precipitated by boiling with phosphoric acid, and the chlorides of lithium and manganese are left in solution. Various methods have been proposed for the removal of the manganese from this solution, but barium sulphide appears to be as suitable as any. Any barium in solution

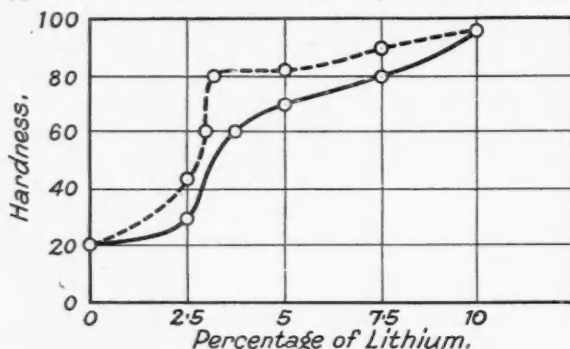


FIG. 2.—THE HARDNESS OF LITHIUM-ALUMINIUM ALLOYS.

can be removed in the usual way, and the clear, filtered solution treated after concentration with potassium carbonate; or, alternatively, it may be treated with oxalic acid to precipitate lithium oxalate, from which the carbonate may be obtained by gentle ignition.

### The Metal

The methods available for the production of metallic lithium are limited to those involving electrolysis, either of a fused salt or of a solution of one of the salts in an inert solvent such as pyridine. The early attempts at the preparation of metallic lithium were made with lithium chloride, and were attended with moderate success. Later experiments have shown that the bromide and iodide offer a better method for its production. Large-scale experiments have shown that a mixture containing lithium bromide with 10 to 15 per cent. of lithium chloride and 20 per cent. of potassium chloride gives very good results. This mixture is fused in large graphite crucibles which form the anode, the cathode taking the shape of a hollow iron cylinder. The metal separates quite readily on the passage of the current, but difficulties arise in removing it; it rises to the surface of the pot and short circuits the current, and on account of its low melting point has to be removed in the liquid state.

These difficulties can be overcome by the use of a more modern type of cell in which the electrodes are separate from the container, which is heated by producer gas. They pass through gas-tight covers, so that the chlorine produced during electrolysis can be removed. The cathode is surrounded by a gauze cylinder, inside which the liquid lithium collects, being unable, on account of its high surface tension, to pass through the gauze. It can be removed from time to time by means of a special covered ladle.

It is also possible by means of the electrolysis of a solution of lithium chloride in pyridine to obtain a coherent mass of lithium by electrodeposition, but there is no evidence to show that this process has attained any commercial importance.

### Properties

When freshly cut, lithium has the silvery appearance which one usually associates with sodium, but this lustre soon becomes tarnished on exposure to air, although lithium is very much less oxidisable than the other alkali metals; further, it is not so soft as these metals, although softer than lead. Its melting point is  $180^{\circ}\text{C}.$ , and it has the property of being the lightest of all solid substances at ordinary temperatures. Its density is given variously as 0.59 to 0.60.

Obviously, such a metal has no structural value *per se*, but it is an attractive substance with which to experiment on the production of ultra-light alloys; for instance, an alloy of magnesium with 30 per cent. of lithium has a density of 1.4, and, could it be obtained strong enough, would prove of the utmost value. As it is, lithium finds use only in alloys which contain it in small percentage.

Lithium dissolves easily in aluminium, but the alloys containing more than 10 per cent. of lithium have no chemical stability. The eutectic point lies at the position corresponding to the formation of a compound  $\text{Al}_3\text{Li}_2$ , and contains 14.5 per

cent. of lithium. Fig. 1 gives the phase diagram of aluminium and lithium.

The effect of the presence of lithium on the hardness of aluminium is shown in Fig. 2; the continuous line indicates the hardness of the untreated alloy and the dotted line the hardness of the quenched and aged alloy. The quenching took place from  $500^{\circ}\text{C}.$ , and a considerable increase of hardness is noted; the hardening effect of lithium is enhanced by the presence of copper in the aluminium. Thus an alloy of aluminium containing 2 per cent. of copper and 0.4 per cent. of lithium has a Brinell hardness of 8.4 after quenching from  $500^{\circ}\text{C}.$ , and ageing at  $18^{\circ}$  to  $100^{\circ}\text{C}.$  Lithium has a stronger hardening effect than has magnesium, but Corson remarks that it is no cheaper in use than the larger amount of magnesium required to produce the same effect, and that even in the presence of manganese, nickel or copper the hardness will not go above 100 Brinell, nor the tensile strength above 60,000.

In spite of the above statement, there appears to be a definite advantage in using lithium as an age-hardening and promoting factor; the Scleron and Aeron alloys are made with lithium as a hardener. They contain about 85 per cent. of aluminium, the remainder being composed of copper, nickel and zinc, with small quantities of manganese, silicon and lithium. The melting point is approximately  $600^{\circ}\text{C}.$ , the specific gravity 2.8 to 3.0, and the limit of elasticity 10 to 20. So far, lithium metal is only at the commencement of the period of its useful application, and it will be of interest to observe the future developments associated with it.

### The Compounds of Lithium

The chloride and nitrate of lithium are very deliquescent substances, which are seldom met with, and the most common salt of lithium in ordinary chemical practice is the carbonate. This substance, in contradistinction to the carbonates of the other alkali elements, is only sparingly soluble in cold water, and has a very abnormal solubility curve (shown in Fig. 3). It is sufficiently soluble in water at the ordinary temperature to give a solution which is valuable in the "blueing" of microscopic sections of animal material to illustrate cell-structure. The blueing is caused by the alkaline nature of the lithium carbonate solution; the section is previously stained with haemalum.

In industry, the fluoride and fluophosphate of lithium are used in ceramics for opacifying certain types of glass, and the addition of a soluble lithium salt to the acid of storage bat-

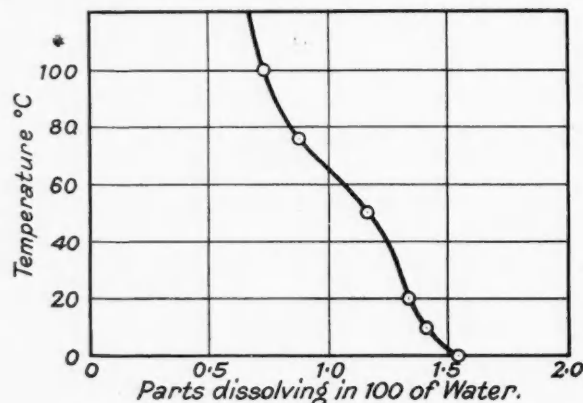


FIG. 3.—THE SOLUBILITY OF LITHIUM CARBONATE.

teries is said to prevent the harmful influence of absorbed carbon dioxide. The main use of lithium salts is, however, in pharmaceutical practice, where it is often used in the preparation of medicines for the alleviation of gout. There is quite a mass of evidence to show that lithium has some alleviating influence, although the theoretical considerations are not so clear. The beneficial action is supposed to arise from the fact that the urate of lithium is very much more soluble in water than the other urates of the alkali metals, and hence it has been suggested that the administration of lithium will lead to the formation of lithium urate and so diminish the uric acid deposits at the gouty joints.

## Metallurgical Topics: Monthly Notes and Comments

### From Our Own Correspondents

#### Chemically Pure Iron

WHILE commercially pure iron is now cheaply produced in large quantities, chemically pure iron, and still more so spectroscopically pure iron, appear to evade the investigator. Final traces of carbon cling most pertinaciously, and, as Yensen has shown in a paper of extreme interest, "On the Road to Pure Iron and Some of Its Indicated Properties," it has proved almost impossible up to now to divest iron of the element that more than any other masks its real and ultimate properties. The paper referred to was read before the American Electro-Chemical Society at its Pittsburg meeting on September 19. Iron can now be readily produced of a purity of 99.95 per cent. The remaining 0.05 per cent. of impurities consist partly of interstitial elements which insert themselves into the iron lattice and strenuously resist removal, and partly of substitution elements which it is more easy to get rid of. Carbon and phosphorus remain interstitially, and there is evidence that, even at room temperature and after extremely slow cooling, 0.005 per cent. of carbon remains in solution in iron, while at 700° C. the solubility is of the order of 0.05 per cent. Oxygen, likewise, is an interstitial impurity, sparingly soluble, but ejected only with great difficulty from the iron lattice.

#### Elimination of Carbon and Oxygen

THE removal of carbon and oxygen down to 0.01 per cent. each offers no great difficulty, and methods for dealing with the latter are described by Mr. Yensen. To eliminate 0.01 per cent. of carbon was more difficult, once the amount of that element had already been brought down to a practicable limit. Yet the effect of even so minute a quantity on the magnetic properties of iron is estimated to be enormous. Of pure iron, therefore, we as yet know little. By extrapolation, Mr. Yensen seeks to penetrate some of its mysteries, while frankly admitting that such a method is indecisive. With 0.05 to 0.06 per cent. of carbon present, permeabilities up to 60,000 have been obtained, and indeed permeability may be employed as some measure of the carbon present. Speaking generally, the purer the iron the less complex its actions and behaviour due to external conditions. The view is put forward that pure iron would display no allotropic transformation points; that  $A_3$  and  $A_4$  are due to the presence of interstitial impurities in the absence of which the points themselves would disappear. The paper is a very suggestive one, and, while throwing perhaps no new light on a very difficult problem, summarises clearly the factors needed for its elucidation.

#### Metallurgical Waste

THE amount of scrap made in a metallurgical works bears some rough inverse proportion to the efficiency of the processes employed; in any case, the bulk of it is carefully collected, kept separate, and used again, by remelting. Comparatively little is thrown away; the four ounces of gold recovered from town refuse in Birmingham was derived from some other source than a works, or even a jeweller's workshop. In addition to this, there were notable quantities of copper and brass, zinc and lead, besides the inevitable tin cans, and altogether some £2,000 was "recovered" from the debris of the city in the course of the year. Following thereon there have been fantastic tales of the recoverable values from the "waste" of London, and estimates of meticulous exactitude of the tonnages of various metals likely to accrue—all in the daily Press, which when dealing with technical matters often goes badly astray. The amount of "metal" which it is stated could, in an average year, be recovered from London's domestic waste, is 77,606 tons, and the context implies that it would consist in the main of tin cans. De-tinned and bundled as scrap for steel furnaces, the recovery of all this metal is held out as a profitable source of municipal wealth.

#### Detinning Processes

THERE are a tremendous number of processes patented for the recovery of tin from old tin plate. Very few of them are workable, and still fewer remunerative. To begin with, three pounds of tin per hundredweight of new tin plate is a generous estimate of the original amount of tin present. Barely 50 per cent. is recoverable, even by the best of the processes—the

chlorine process. Nor would much more of the metal waste than 50 per cent. be in utilisable tin cans. This gives a rough estimate of 1,000 tons of tin, the cost of recovering which would be somewhere about £2 per ton of scrap treated, plus the cost of collection and transport. There may, after all, be something in it, but nothing like the optimistic reports which have been appearing of late. As for the methods of recovery which involve the easy, but impracticable, suggestion of heating the scrap up in "shaft furnaces" and merely running the molten tin out at the bottom, most metallurgists who have tried their hand at tin recovery from scrap will be inclined to agree that it simply cannot be done in this way. The alternative, of forgoing the tin and merely bundling the scrap, will not do, for steel makers, rightly or wrongly, object to charging tin into their furnaces. Stead, long ago, adduced evidence to show that the presence of tin in steel may be exceedingly detrimental; the resulting metal neither rolls well nor behaves as well as tin-free steel. Indeed, the object of detinning is as much to meet the objections of users of the scrap as to recover the tin, which is an evasive metal to extract from thin tin plate. It is doubtful whether the metal recovery prospects of the schemes that have been so much discussed are as lucrative as is often stated.

#### German Research on Beryllium

BERYLLIUM has not (as has been stated before in these columns), justified, up to now, all the expectations that some years ago were entertained in regard to it, but it still attracts a very considerable degree of attention and interest amongst metallurgists. At the recent meeting of the Institute of Metals in Germany, many of the members visited the extensive laboratories of Siemens and Halske, at Siemensstadt, where researches on the metal and its alloys have been actively pursued. They have, indeed, been made the subject of a valuable monograph issued by the firm, which supplements the information hitherto forthcoming and the work that has been done elsewhere, notably in America. Much more of the metal is now available, by means of the new method of electrolyzing mixtures of the oxyfluoride and barium fluoride. Beryllium is obtained, by this process, 99.5 per cent. pure, the impurities being chiefly aluminium, carbon and iron. From the purified metal it has been possible to correct or confirm many of its constants. Thus the atomic weight is given as 9.018; the melting point 1,278° C.  $\pm$  50°; the heat of fusion 341 calories/gramme, and the heat of oxidation 140.15. The co-efficient of expansion is nearly that of mild steel; the electrical conductivity about 1/12th that of copper; and the solution potential midway between that of magnesium and that of zinc. It tarnishes easily in air, displays a complete lack of ductility at ordinary temperatures, and can easily be powdered by a hammer. At higher temperatures it is ductile, but owing to its affinity for oxygen difficult to work. It is extraordinarily transparent to X-rays, being 17 times more penetrable than equivalent thicknesses of aluminium.

#### Hardness of Some Beryllium Alloys

TURNING now to the problem of its alloys, direct electrolysis has, with some success, been employed for their production. Aluminium alloys containing a little beryllium are made fairly easily by melting the metals together. Up to now, however, the aluminium-beryllium alloys present few features of interest. The beryllium-copper alloys are possessed of some valuable properties. Beryllium hardens copper markedly, while the ultimate tensile strength of some of the beryllium-copper alloys reaches as high as 95 tons per square inch. Age-hardening can be accelerated, or made to occur at lower temperatures, by the addition of phosphorus to these alloys. Nickel and beryllium alloy easily, and form, at 1,155° C., a eutectic—containing about 5.25 per cent. of beryllium.

Certain alloys of iron and beryllium, and of iron-nickel-beryllium, likewise, display industrially useful properties of hardness, while with chromium present up to 680 Brinell has been obtained, and thin sheets three times as strong as duralumin have been rolled from a beryllium "Invar," which, it is suggested, will find valuable applications in aeroplane and airship construction.

### Segregation in High Carbon Steel

THE crystallisation and segregation phenomena involved in the solidification of comparatively small steel ingots containing high (1.10 per cent.) carbon were the subject of a masterly paper read before the Iron and Steel Institute, at the autumn meeting in Newcastle-upon-Tyne last month. It forms a valuable supplement, so to speak, to the work accomplished so far by the Heterogeneity Committee, which has published two reports dealing with low and medium carbon steel ingots, including nickel steel. The paper referred to was by Mr. Axel Hultgren, and embodies a considerable amount of very careful investigation. What happens during the cooling of one kind of steel, cast into ingots of a certain size, is not, of course, necessarily indicative of what happens in the case of ingots of a different composition, cast in ingots of a totally different size, yet by careful correlation of all the ascertainable factors, such as temperature of casting, time of teeming, thickness and temperature of the moulds, influence of a sink head, top *versus* bottom pouring—or even multiple casting—and composition, and by integration of the results, certain broad principles may emerge. Mr. Hultgren's steels were made in a basic electric arc furnace of 3.5 tons capacity, and top-poured, big end up. A preheated sink head of refractory material was used; in some cases the moulds, too, were slightly warm, and the metal had an average composition of 1.10 carbon; 0.20 silicon; manganese 0.30; phosphorus 0.012 and sulphur 0.009 per cent. Ferro-silicon was added, in the ladle, and aluminium sometimes added in the ladle or in the mould.

### Growth of the Dendrites

THE paper is well illustrated, and the results are conveniently summarised. The first portion of the investigation discusses the formation and ultimate structure of the surface crystals, as revealed by etching away the surface, in increments of a millimetre, to thicknesses of 37, 38 and 39 millimetres respectively, thus revealing the structural track of the individual crystals. Their behaviour when after-pouring is resorted to is also described, and the *rationale* of dendritic growth. The growing surface of an ingot is subject to deforming stresses caused by suppressed contraction of the steel and by expansion of the mould. From this, two results arise; inverse segregation at the surface, and the deformation of the growing crystals. In some steels solidifying as  $\delta$ -iron and subsequently transforming into austenite, a transformation structure consisting of elongated grains is formed, the boundaries curving as the isothermals of the transformation change. The interior usually crystallises from independent nuclei, for which, usually, undercooling appears necessary. Such nuclei grow rapidly into large dendrites, but if stirring is resorted to the interference with quiet cooling results in a small-grained structure. In regard to segregation itself, Mr. Hultgren distinguishes three types—upwards, downward, and sedimentation (both positive and negative in the axial region). Their causes are discussed, and the view put forward that varying the taper of the mould is a method of obviating the occurrence of V-segregation. For this purpose an ingot profile has been designed with a large taper in the middle portion, and little or no taper in the end portions.

### The Institute of Metals

THE Institute of Metals has just issued its programme for the coming session. This includes over 40 meetings to be held in metallurgical centres throughout England, Scotland and Wales. The programmes of the various local sections have been drawn up to meet the needs of local industries. The subjects dealt with are of an essentially practical character, and include papers on "Metals in Aircraft Construction"; "Metal Melting by Electricity"; "Chromium Plating"; "Some Difficulties in Aluminium Alloy Founding and Some Remedies"; "Electric Heat-Treatment Furnaces"; and "The Effect of Some Impurities in Copper." Copies of the programmes can be obtained on application to the Secretary, Mr. G. Shaw Scott, M.Sc., 36, Victoria Street, London, S.W.1.

### Tin Spraying

THE honorary secretary of the Tin Industrial Applications Committee, a body recently formed in co-operation with the British Non-Ferrous Metals Research Association to investigate the industrial adaptability of the metal, announces that the spraying of tin will be among the subjects of investigation.

He stated that it was claimed for the process that pure tin could be sprayed successfully on to any surface, from steel and glass to wood or silk. The first concern of the Committee would be to verify the results of recent experiments that went to show that pure tin sprayed by the process upon a metal structure rendered it proof against corrosion. Since it had been stated that corrosion cost the heavy industries some £500,000,000 a year, the matter was deserving of careful consideration. This method of spraying tin might open up a new era in the manufacture of decorated fabrics for women's clothes and furnishing. Tin could be sprayed upon materials as delicate as silk, which afterwards, it was claimed, could be crumpled and washed without damaging the fabric or the metal design. Tin sprayed upon glass would make satisfactory and cheap reflectors and possibly mirrors, while novel effects in interior decoration could be obtained. There would be a wide use in tin-spraying the interior of tanks and receptacles used in the manufacture of food. It was the business of the Committee to examine any new process which they considered of commercial interest.

### The Ebbw Vale Steel Works

SIR FREDERICK MILLS, managing director of the Ebbw Vale Steel, Iron, and Coal Co., Ltd., had an interview with Mr. J. H. Thomas, Lord Privy Seal, on Monday, regarding the company's decision to close its iron and steel works, thus throwing thousands of workers out of employment. Sir Frederick Mills has been making strenuous efforts to save the works, the closing down of which would mean the ruin of a town of 30,000 people. Notices have been served on many men, the reason being that the firm is seriously affected by the price-cutting of Continental firms. Sir Frederick Mills was urged by Labour leaders in Ebbw Vale and others to take action in this matter. Accordingly he travelled to Brighton to see Mr. Thomas, who was attending the Labour Party Conference. Mr. Thomas afterwards stated that he had had an interview with Sir Frederick Mills, who had placed all the facts before him, and he was now considering the whole situation.

The interest which Mr. Thomas takes in the ferrous industries is further shown by his recent interview with a deputation of iron and steel manufacturers, immediately after his return from Canada. After the interview the following official statement was issued from Whitehall:—"The Lord Privy Seal, Mr. J. H. Thomas, met to-day (Thursday, September 26), representatives of the National Federation of Iron and Steel Manufacturers from all sections of the iron and steel industry. He discussed with them the possibilities of the Canadian market for the products of the British iron and steel industry in the light of his recent visit."

### The Value of Geological Surveying

How a gold prospector found and left behind the world's largest ore deposit was described by Sir Albert Kitson in his presidential address to the Geological Section of the British Association at Johannesburg recently.

The prospector had sunk a shallow hole on the side of a hill and unearthed good manganese ore, but, not recognising its identity and probably regarding it as iron slag, he apparently took no special notice of it. While the director of the geological survey was surveying the Insuta manganese ore deposit after his discovery of it, he found this old hole, and noted that the prospector had failed to discover what later proved to be one of the largest and richest deposits of manganese ore in the world—one that was of great importance to the life of the British nation at a most critical period of the Great War. Another case instanced by Sir Albert was also of a prospector for gold who sank a shaft over 40 feet deep through bauxite—a valuable mineral used in the manufacture of aluminium. He used the shaft constantly without knowing, until informed of the fact by the same geologist, that the material he had excavated was bauxite. In both cases the want of geological knowledge was the cause of the failure of the prospectors to recognise what they had excavated.

The title of the paper was "The Utility of Geological Surveys to Colonies and Protectorates of the British Empire." In giving a detailed list of the functions of a geological survey, the speaker included "Advice to Government regarding operations of prospectors and prevention of fraudulent flotation of companies."

## Some Inventions of the Month

By Our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature, published weekly in THE CHEMICAL AGE.

### Copper

SPECIFICATION 318,314, dated June 5, 1928, by T. J. Taplin, B. Taplin, and Metals Production, Ltd., of London, describes various improvements in the process of Specification 250,991 (See THE CHEMICAL AGE, Vol. XV, p. 7 [Metallurgical Section]), according to which oxidised ores of copper are heated in admixture with solid carbonaceous matter and in presence of available halogen at a temperature below the melting point of copper and yet adequate to produce a reaction which separates the copper from the gangue by segregating it to the outside of the ore particles. It is now found that a substantial economy can be effected by subjecting the gases drawn from the segregating reaction to treatment for recovering their content of available halogen. Thus the halogen may be recovered by absorption from the gases by limestone, or a copper carbonate ore, or by scrubbing the gases with water. In any case, the resulting halides are utilised as a source of available halogen for the treatment of further quantities of the ore in the segregation furnace.

### Chromium Plating

THE deposition of chromium on the cathode is facilitated, and a more even deposit is obtained, by the use of an anode of aluminium in place of the steel usually employed. Such an anode is preferably in the form of a plate, casting, grid, cage, or the like of aluminium, or of a cage or receptacle containing powdered, granulated, or broken aluminium, and requires to be deoxidised periodically, *e.g.*, by washing, dipping into a 20 per cent. solution of caustic soda, brushing, and again washing. The preferred electrolyte is an aqueous solution of chromic acid of 25–30° Bé. See Specification 317,137, dated May 15, 1928, by C. H. R. Gower and Stafford O'Brien and Partners, Ltd., of London.

### Extracting Metals

THE extraction of metals such as lead, cadmium, bismuth, silver, gold, and antimony from their compounds, especially from their oxides, is effected by reduction with arsenic trioxide at 400–650° C. in a fused alkali metal compound such as caustic soda. The alkali arsenate separated from the melt may be converted into calcium arsenate with recovery of caustic alkali for further use. Examples are given of the treatment of dusts containing as main constituents (1) arsenic and antimony oxides, (2) antimony oxide and cadmium, (3) arsenic and lead oxides. See Patent Application 315,811, bearing the International Convention date July 18, 1928, by G. N. Kirsebom, of New York.

A PROCESS for recovering base metals and/or salts thereof (other than aluminium salts) from ores or materials containing such metals other than tin is described in Specification 318,301, dated April 5, 1928, by E. A. Ashcroft, of Ashburton, Devon. The invention is of very wide application, but is described mainly with reference to the recovery of lead, zinc, and copper. A mixture of the ore, etc., with dry ammonium chloride is subjected to a carefully regulated heat treatment in order to form desirable salts of the metals and to liberate ammonia, usually in association with carbon dioxide and/or sulphur. This ammonia is then used for precipitating, separating, modifying, or recovering the metals or salts with regeneration of ammonium chloride for use again. The principles underlying the various steps of the process, and its application to a wide range of ores, concentrates, and residues, are discussed in considerable detail, and an example is given of the detailed procedure adopted for extracting the zinc and lead from a material containing 63.95 per cent. of zinc (as oxide), 11.55 per cent. of lead (as oxide), and 0.20 per cent. of copper (as oxide), together with some iron, cadmium, and tin.

### Iron

A PROCESS for producing molten iron or steel directly from the ore and controlling its sulphur content is described in Patent Application 316,303, bearing the International Convention date, July 28, 1928, by Davis Steel Process Corporation of New York, assignees of E. W. Davis, of Minneapolis, Minnesota, U.S.A. A mixture of ore, carbon, and fluxing

material is heated in a reverberatory furnace until the ore is reduced, and the temperature of the furnace is then maintained such that the reduced metal is carbonised at a rate sufficient to prevent its oxidation. Thus the iron oxide content of the slag is maintained sufficiently low and removal of the sulphur from the metal is facilitated.

A MALLEABLE cast iron is obtained by heating white cast iron to a temperature, *e.g.*, 1,000° C., high enough to cause the cementite to go into solid solution, maintaining this temperature for a few hours, cooling to 675–750° C., maintaining this temperature long enough (8–36 hours) for the iron to become malleable, and cooling. The heating may be effected electrically, or in a bath of a molten salt such as barium chloride, or in a vacuum furnace. See Patent Application, 315,724, bearing the International Convention date July 16, 1928, by British Thomson-Houston Co., Ltd., of London, assignees of I. R. Valentine, Erie, Pennsylvania, U.S.A.

### Iron Alloys

SPECIFICATION 317,998 (Johnson), a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany, dated August 31, 1928, describes a process for obtaining iron alloys from pulverulent initial materials by heat treatment without melting. A pulverulent mixture of iron and/or iron oxide, prepared from iron carbonyl and alloy constituents wholly or partially in the form of oxides or other reducible compounds, is subjected to a heat treatment under reducing conditions without melting. The treatment is preferably effected at 800–1,000° C., and it is found advantageous to apply a pressure treatment before, during, or after the heating. Specified alloying metals are chromium, manganese, vanadium, molybdenum, tungsten, cobalt, and nickel, and the process eliminates the difficulty experienced in obtaining such metals in the finely divided state necessary for the direct production of homogenous alloys. The pulverulent iron may be obtained by thermal decomposition of iron carbonyl, as described in Specification 269,677 (See THE CHEMICAL AGE, Vol. XVI, p. 534), and the iron oxide by oxidation of such pulverulent iron or by burning iron carbonyl as described in Specification 258,313 (See THE CHEMICAL AGE, Vol. XV, p. 402).

### Reduction of Ores

THE reduction of iron ores by a method which is stated to be applicable to the treatment of ores containing chromium, nickel, manganese, or copper, is described in Patent Application 315,760, bearing the International Convention date, July 17, 1928, by Davis Steel Process Corporation of New York, assignees of E. W. Davis, of Minneapolis, Minnesota, U.S.A. A mixture of ore, carbon, and slag-forming materials is heated in a reverberatory furnace to such a temperature (1,500–1,575° C.) that the reduced metal is carbonised by the carbon, converted to graphitic form, at such a rate that any oxidation of such metal is avoided. By introducing a suitable excess of carbon with the charge, a protective layer of carbon-containing gas is maintained on the surface of the bath to prevent oxidation and effect the carbonisation.

### Refining Metals and Alloys

REFINING processes dependent on the physico-chemical action of slags on metals and alloys in the liquid state have certain disadvantages and limitations, which are discussed in detail in Specification 317,180, dated June 25, 1928, by S. Westberg, of Oslo, Norway. In accordance with the invention, castings or ingots are obtained from ferrous or non-ferrous scrap metal or solid melting stock by refining the metal in a furnace by a gaseous reagent at a temperature below liquefaction, but above 700° C, without the presence of, or contact with, any solid or liquid slag, and thereafter melting in the same or a separate furnace. The gaseous reagent may be a mixture of hydrogen and an inert gas such as nitrogen, with or without the addition of water vapour, or may be a suitable hydrocarbon or a mixture of hydrocarbon and hydrogen, with or without an inert gas. The refining stage of the process is preferably effected under a pressure of about 10 atmospheres, and the subsequent melting stage at a pressure below atmospheric. A further refining by gaseous reagents may be effected during melting, and a final purification of the ingots or the articles made therefrom may be effected by heat treatment and exposure in solid form to gaseous reagents.

## Current Articles Worth Noting

*We give below a brief index to current articles in the technical Press dealing with metallurgical subjects.*

**ALLOYS, FERROUS.**—On the equilibrium diagram of the iron-molybdenum system. H. Takei and T. Murakami. *Transactions Amer. Soc. Steel Treating*, September, pp. 339-371.

**ANALYSIS.**—The determination of calcium and magnesium in aluminium, in the simultaneous presence of silicon, iron, copper, zinc, lead, manganese and titanium. K. Steinhäuser. *Zeitschrift analytische Chem.*, Vol. 78, Part 5-6, pp. 181-188 (in German).

The determination of manganese in steel according to Wald. J. Kassler. *Chemiker-Zeitung*, September 14, p. 719 (in German).

**CASE-HARDENING.**—A new method of testing the depth of case on carburised steel. F. A. Firestone and E. J. Abbott. *Metals and Alloys*, Vol. 1, No. 1, July, p. 18.

**CORROSION.**—The effect of depolarisation on the rate of rusting and its practical application. F. Tödt. *Korrosion und Metallschutz*, August, pp. 169-174 (in German).

The corrosion and rusting of unalloyed and alloyed cast iron. P. Köttschke and E. Piwowarsky. *Korrosion und Metallschutz*, August, pp. 174-176 (in German).

The prevention of corrosion. E. S. Stokes. *Chem. Eng. and Mining Rev. (Australia)*, August 5, pp. 432-440.

**GENERAL.**—Demands of chemical industry on metallurgy. B. D. Saklatwalla. *Metals and Alloys*, Vol. 1, No. 1, July, pp. 8-13.

The origin of the structure of cast metals. G. Tammann. *Zeitschrift Metallkunde*, September, pp. 277-282 (in German).

Technical problems in the solidification of metals. G. Masing. *Zeitschrift Metallkunde*, September, pp. 282-286 (in German).

The shrinking of metals. F. Sauerwald. *Zeitschrift Metallkunde*, September, pp. 293-296 (in German).

The centrifugal casting process. H. Simon. *Zeitschrift Metallkunde*, September, pp. 302-304 (in German).

Bearing metals on a lead, antimony and tin basis. H. Müller. *Zeitschrift Metallkunde*, September, pp. 305-310 (in German).

**HEAT-TREATMENT.**—Proposed control methods for small heat-treatment plants. W. J. Crook. *Transactions Amer. Soc. Steel Treating*, September, pp. 414-428. The author shows that a hardness-testing instrument and the use of a furnace are adequate equipment with which a small heat-treatment plant may obtain considerable information about its raw materials and products.

**METALLIC COATINGS.**—Investigations on cadmium coatings. R. Hopfelt. *Korrosion und Metallschutz*, August, pp. 176-180 (in German).

**PASSIVITY.**—The theory of passivity phenomena. VI. The passivation of chromium at low current densities. W. J. Müller and K. Konopicky. *Monatshefte*, Vol. 52, Part 4, pp. 289-296 (in German).

**STEEL.**—The transformation of austenite into martensite in hardened steel. E. Scheil. *Zeitschrift anorganische Chem.*, Vol. 183, Parts 1-2, pp. 98-120 (in German).

A study of the burning and overheating of steel. II. W. E. Jominy. *Transactions Amer. Soc. Steel Treating*, September, pp. 372-392. The results are summarised in a table showing the temperatures at which a series of plain carbon and alloy steels will burn if forged immediately after heating in a direct gas-fired furnace when using an excess of gas and air.

Effect of annealing on the grain structure of extremely hard-rolled steel ribbon. N. P. Goss. *Transactions Amer. Soc. Steel Treating*, September, pp. 405-414.

**STEEL, ALLOY.**—Maurer's manganese steel in the development of rustless steels. E. Maurer. *Stahl und Eisen*,

August 22, pp. 1217-1220 (in German). The work of Brearley and Haynes; Borchers' metal; priority of the German work; the steels of Guillet and Strausz; the work of Monnartz; and the Monnartz-Borchers' patent; the publication of Friend, Bentley and West; the first recognition of rustless steel, by Maurer, on September 5, 1912; the addition of nickel; the incorrect views of Monypenny.

**STEEL, HIGH-SPEED.**—Salt baths for the hardening of high-speed steel. B. Kjerrmann. *Transactions Amer. Soc. Steel Treating*, September, pp. 393-403. A chamotte crucible, as the container for barium chloride to which a sufficient amount of ferrosilicon was added, could be used with no risk of decarburising high-speed steel and with an increase in the life of the crucible of about 4.5 times that obtained with moulded silica crucibles.

A new furnace for heat-treating high-speed steel. W. B. Hall. *Transactions Amer. Soc. Steel Treating*, September, pp. 399-404. A new electric salt bath, now in extensive use in the United States, is described. The bath is heated by current flow through the bath itself, from an immersed electrode in the pot wall.

**URANIUM.**—The preparation of pure uranium. E. Botolfsen. *Bulletin Soc. Chimique France*, July, pp. 626-627 (in French). Uranium oxide,  $U_3O_8$ , is heated with sublimed metallic calcium in an electric furnace *in vacuo*. After separation of the calcium oxide by passage through a sieve (the uranium, being in the form of a fine powder, passes through), the uranium is treated with cold 2 per cent. acetic acid, washed with water, and dried with alcohol and ether. Analysis indicates 99.95 per cent. purity, and X-ray analysis shows no trace of impurity.

## Commercial Intelligence

*The following are taken from printed reports, but we cannot be responsible for any errors that may occur.*

### Mortgages and Charges

[NOTE.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debts due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case, the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.]

**GALLOIS LEAD AND ZINC MINES, LTD.**, London, E.—Registered August 27, £500 debentures, part of £20,000; general charge (excluding plant, etc., where purchase price is not fully paid).

**LYSAGHT (JOHN), LTD.**, Bristol, ironfounders.—Registered August 24. Trust Deed dated August 14, 1929, securing debenture stock for £2,500,000 and premium of 3 per cent.; general charge. \*£124,007. October 3, 1928.

**NEPTUNE FOUNDRY CO., LTD.**, Liverpool.—Registered September 6, £1,900 debentures; general charge. \*£2,000. February 28, 1929.

**YEADON, ADNITT AND CO., LTD.**, Bristol, ironfounders. Registered September 9, £750 debentures; general charge (ranking in priority to £1,500 debentures, dated May 14, 1925). \*£2,750. April 5, 1929.

### Satisfactions

**BLAENAVON CO., LTD.**, coal and iron masters.—Satisfaction registered September 18, £8,600, part of amount registered August 24, 1911.

**NEPTUNE FOUNDRY CO., LTD.**, Liverpool.—Satisfactions registered September 6, £1,000, registered May 27, 1922, and £900, balance of amount registered March 14, 1928.

**SHELTON IRON STEEL AND COAL CO., LTD.**, Stoke-on-Trent.—Satisfaction registered September 10, £184,600, outstanding July 1, 1908.





# Monthly Metallurgical Section

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**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

## The Fabrication of Acid-Resisting Steel Plant

By Dr. W. H. Hatfield

A lecture on the above subject was delivered by Dr. Hatfield before the Institution of Chemical Engineers on Friday, October 25. An account of the main points of the lecture appears below.

DURING recent years great advances have been made in the production of acid-resisting steels, by the addition of chromium, nickel and other elements. A survey of the technical literature and the patent files will disclose an extremely wide range of composition of steels which are now put forward as being suitable for the manufacture of chemical plant and apparatus. Since, however, the author is particularly dealing with the problems arising in the utilisation of such steels, it will be appreciated that he can best serve the immediate purpose by confining himself to the range of composition which actual practice has shown to be most eminently suitable, and hence utilised on a large commercial scale.

The first step in the progress in this field was naturally the production of the plain chromium steels containing approximately 13 to 14 per cent. of chromium. Such steels, however, have only a limited range of resistance when compared with the class of steel with which it is proposed to deal, and, furthermore, since they are martensitic (*i.e.*, air-harden when cooled quickly after heating in excess of the critical range), a limitation is set to ease of fabrication from several points of view. The range of composition of the steels with which the author is proposing to deal in the present instance covers a chromium content of 14 to 20 per cent., a nickel content of 7 to 12 per cent., with a carbon content not exceeding 0.20 per cent. Steels of such composition are classed as austenitic steels—*i.e.*, they consist of a solid solution of carbides in a matrix consisting of iron containing chromium and nickel in solution—and therefore are fundamentally different from the martensitic class of steels previously referred to. The mechanical properties of these steels are indicated in table I.

The factors influencing design of a particular plant are: (1) convenience and suitability for the work to be done; (2) cost and ease of fabrication; (3) strength and rigidity.

Strength and rigidity need to be considered separately. In large vessels, pans, etc., under light pressures the stresses to be carried by the material are relatively small, but adequate thicknesses must be provided for the vessels to retain their shape in construction and transport as well as in use. In some cases very thin-walled vessels suitably strutted or corrugated may be used. In other types of plant heavily loaded or under high pressures, dimensions must be such as reasonably to withstand the stresses imposed. A design which is satisfactory in mild steel in these respects will generally be suitable with only slight modifications in softened austenitic nickel-chromium steel. Some slight reduction is possible in many cases, owing to the fact that loss of thickness by corrosion has not to be allowed for where austenitic nickel-chromium steel is suitable. In replacing copper or aluminium, softened austenitic nickel-chromium steel of smaller thicknesses is generally suitable, but rigidity must not be overlooked.

### Heat Transmission

With regard to vessels for heat transmission, austenitic nickel-chromium steel differs from mild steel in having a lower thermal conductivity and a higher thermal expansion. Thermal conductivity naturally affects the "rate of heat transmission," but to a degree which has little relation to the actual thermal conductivity values. Even compared with copper, which has thirty times the conductivity, the difference in heating rate varies from nothing to only  $\frac{1}{3}$  or  $\frac{2}{3}$ , according to circumstances. The differences are least where the whole

Table I

Material.	C.	Mn	Si	Cr	Ni	As Rolled Condition.	Y.P. tons. sq. in.	M.S. tons. sq. in.	Elong. Per cent.	R. of A. Per cent.
Martensitic Stainless "Iron" .....	11	19	21	14.8	23	Air-hardened and tempered 975/750°	38.8 24.2	63.0 30.1	12.0 29.6	29.5 70.2
Austenitic 15/11 .....	13	25	30	15.2	11.1	Softened 1150° C.	24.6 16.8	43.2 36.8	41.0 51.8	39.0 50.0
Austenitic 18/8 .....	12	24	32	18.1	7.9	Softened 1150° C.	27.5 17.3	49.8 43.0	39.5 48.0	39.0 46.0

Each particular steel has its range of chemical resistance, which may be modified by the addition of special elements such as molybdenum, tungsten, copper, etc., but it will be realised that when producing in bulk it is desirable not to multiply different compositions, and it is therefore not surprising that the more complex steels just referred to are only produced when required for specific purposes.

The materials which have been extensively employed in plant manufacture are those containing either 18 per cent. chromium and 8 per cent. nickel, or 15 per cent. chromium and 11 per cent. nickel. The observations, therefore, in the following pages refer particularly to steels of such compositions.

exterior of the vessel is heated, as in steam-jacketed pans. If, however, local heat only is applied, as by a gas burner, temperature differences in the austenitic nickel-chromium steel walls are greater than with copper, etc., and this affects the amount of heat transmitted. This is very marked in thin-walled vessels. Thermal expansion effects must be provided for, as severe local heating causes local expansion of a high order, which may lead to warping, etc. Local stresses of an alternating character due to this cause may actually lead to cracking, unless allowed for.

It is frequently necessary to shape steel by deforming whilst hot, and this is a field in which very great care has to be exercised, and it also, at times, calls for somewhat of a com-

promise, since whilst it may be recognised that there is a best range of temperature for such hot working operations, the fact remains that in practice it is not always possible to conform to those conditions. The author's experiences proves that, at times, a knowledge of the importance of the heating range is not appreciated, with unfortunate results. In the production of a steam-jacketed pan, the lower portion of which was hemispherical in shape, pressing was done in four operations whilst at temperatures ranging from 800 to 900° C. No final softening was given before the pan was completed and put into service, with the result that it did not show a proper resistance and actually developed a system of small cracks as a result of the combination of stresses and the bad condition in which the material was left.

A similar result was experienced with another pan which developed cracks after a few months' working, owing to the fact that it had been produced by hand as a result of deformation after numerous heatings on a smith's hearth. Even so, had the pan been fully softened after heating, it would have behaved satisfactorily. The same fabricator made a replace pan, had it properly softened and put into service, where it is still perfectly satisfactory after two or three years' work.

It should always be remembered that these austenitic steels, consisting as they do of a solid solution, should not be subjected to temperatures which tend to break down that solid solution. The range of temperature extending from 500 to 900° C. should be avoided, since exposure to such temperatures tends to precipitate a second constituent along the grain boundaries, which thus destroys not only the chemical resistance, but the mechanical ductility of the material.

#### **Cold Working**

The success of cold pressing of austenitic nickel-chromium steels depends largely upon the press plant and the facilities for softening which are available. Generally the process does not present much difficulty except on medium and large plant where, say, for dishing, the pressing has to be done progressively circumferentially. An interesting example of what can be achieved with due care, without intermediate softening, is a dished bottom made from a  $\frac{3}{8}$  in. plate in sections under a hydraulic press, with a single pair of suitably contoured dies of approximately 15 in. diameter. The dimensions of the dished bottom were 3 ft. diameter by 7 in. deep. This was equal to anything which had been done in mild steel by the people in question at the time.

Cold pressing operations on lighter work on the whole do not present any difficulties. A ratio of diameter to depth of  $2\frac{1}{2}$  or 3 to 1 is easily attainable if the plant has reasonable power. When cold pressing operations are employed it is essential, in the first place, that the steel be fully softened by heating to 1,150° C., and that it is given a satisfactory softening between the different cold-working operations.

#### **Welding Operations**

The process of welding plays an extremely important part in the production of chemical plant from these special steels, and in view of the different methods of welding which are available, it is important that the mechanism of the operations should be carefully reviewed. The following points will appear to any technologist engaged in this field as of fundamental importance:—(1) The necessity for skill and resourcefulness on the part of the operator to avoid warping, etc.; (2) the undesirability of attempting to join thick and thin sections, and the desirability of making the change of section as gradual as possible; (3) stresses of considerable magnitude may be left in the weld owing to the contraction due to the cooling, and particularly the unequal cooling of the hot zones. A subsequent softening operation releases these stresses; (4) if the welding of austenitic chromium-nickel steel to a dissimilar metal is attempted, then particular attention should be given to the difference in the expansions of the two metals.

The processes available for welding operations are either one of the several electric processes or the gas-welding process. The author's experience chiefly relates to the more popular electric processes and the oxy-acetylene process. The electric methods are really to be preferred where possible, and particularly for welding thicknesses in excess of  $\frac{1}{8}$  in. The advantage of the electric method over the oxy-acetylene is that the area heated during welding is more local, and therefore the residual internal stress is less. With the oxy-acetylene process, also, there is a danger of increasing carbon content.

The success or otherwise of welding is often governed by questions of design. As in other branches of engineering, there is frequently insufficient contact between the drawing office and the welding staff. This was particularly noticeable in a recent case in which it was actually expected that 16-gauge material should be welded to  $\frac{1}{2}$  in. thickness of plate. As this was found to be impracticable, a discussion resulted in the alteration of the design so that the local thickness of the thick portion was reduced to  $\frac{3}{8}$  in., making welding to the 16-gauge material feasible and practicable.

The high coefficient of expansion tends to cause trouble when welding sheet material to form rectangular tanks, and allowance should be made for this. If the welding proceeds without a hitch, the tank may finish reasonably free from buckles, but if the welder has erred in several places, there is a likelihood of the tank being severely buckled.

Much trouble is caused through attempting to weld metal which is already severely stressed, and this should be avoided, unless resort is made to subsequent softening. It cannot be emphasised too strongly that, wherever possible, welded articles should be re-softened prior to being put into service.

#### **Riveting**

Hot riveting with these special steels has been conducted on a very extensive scale, and very successfully. The temperature at which the rivet is manufactured should be within the range 1,200 to 1,050° C., and when the rivet is closed it is desirable to work at temperatures approximating to these. The rivet should not be permitted to become scaled during the heating process, since the scale may work out during service and produce leaks.

Cold riveting can be successfully accomplished providing that the rivet is in a properly softened condition, but as the material work hardens fairly rapidly, the operation should be mechanically clean and precise.

#### **Machining**

The machining of these steels does not present special difficulty, provided reasonable precautions are taken, and slight modifications, compared with ordinary steels, are made with regard to shapes of tools, speeds, feeds, etc. The machines and tools used for machining the austenitic chromium-nickel steels should be rigid and free from backlash, the tools being made of good quality high-speed steel. In general, lubricant is to be recommended for machining operations. To turn the steels satisfactorily and obtain a good finish, a keener tool is necessary than when turning ordinary steels.

#### **Brazing and Soldering**

The brazing and silver soldering of austenitic nickel-chromium steel joints are not very extensively employed because (1) the temperatures necessary for these operations usually lie within the range which is unsuitable for the metal, and (2) the evidence tends to show that stress cracks and intercrystalline penetration of the metal by the brass or solder are likely to occur if the metal is in a stressed condition.

The technique of brazing is not easy, as it is essential to protect the parts from oxidation during heating. Blue borax has been found to be superior to ordinary borax as flux, as it contains less water of crystallisation and, therefore, stays in position better. The silver soldering operation has been simplified somewhat by the introduction of a potassium fluoride-boracic acid flux.

#### **Softening**

Mention has been made on several occasions of the desirability for softening. Perhaps it would be well to enlarge on this phase of fabrication. The necessity for softening arises from operations which involve deformation either hot or cold, e.g., hot pressing or flanging, cold spinning, pressing, etc., and where practicable, after welding. The operation simply consists of heating the material or parts to a high temperature and cooling relatively quickly either in the open air, or, where the dimensions and form of the article permit, by quenching in a water bath. The degree of softening obtainable increases with the softening temperatures, but at the very highest temperatures excessive scaling, distortion and very rapid crystal growth occur, and, of course, the higher temperatures are more difficult to attain, so that a compromise is frequently made and the heating temperature is chosen according to the degree of softening required, usually in the neighbourhood of 1,050° to 1,100° C. Experience has

shown that apart from the difficulty of attaining the necessary temperature, the softening of austenitic nickel-chromium steel articles and plant presents very little trouble.

#### Descaling

The removal of scale, incurred in rolling, heat-treatment, and other works' operations, is a very necessary and important section of the manufacture of acid-resisting steels and plant, involving, as it does, huge acid tanks, neutralising and washing plant and efficient drying arrangements. Austenitic nickel-chromium steel, in the course of the manufacture of the sheet, often passes in and out of the descaling plant before the final material is ready. With the present knowledge of scale removal, complete descaling of this class of material can be carried out with comparative ease, and in a very short time. The following conditions are recommended for efficient descaling of these steels:—

#### COMPOSITION OF DESCALING BATH.

Water .. .. .	50 parts by volume.
Hydrochloric acid .. .. .	50 " "
Nitric acid .. .. .	5 " "
Restrainer .. .. .	0.25 " "

The restrainer is one of a class of organic compounds which inhibit attack on the metal without having any material effect on the removal of scale. With the above descaling solution, operating at a temperature of 60° C., complete descaling is obtained after a few minutes' immersion with very little loss of metal. The loosened scale is easily removed by washing with water and scrubbing with a soft brush, after which it is advisable to immerse the steel in a neutralising bath of soda solution, subsequently washing and allowing to dry.

When the descaling of chemical plant is concerned as distinct from the material in its different forms, it is sometimes found advantageous to dilute, in different degree, the strength of the descaling acid.

#### Castings

The whole range of acid-resisting steels can be successfully made into castings of all reasonable shapes and dimensions. The heat-treatment of such castings should conform, on general lines, with the heat-treatment accorded to other forms of the material. Contrary, however, to the conception of many people, no steel casting is ever entirely free from minor forms of shrinkage effects, and this is borne out by the fact that complete soundness in steel ingots, the simplest form of cast shape, is obtained only with the very best technical practice. The chemical engineer will be the first to recognise that perfect freedom from defects is necessary under certain service conditions if absolute freedom from contamination is essential. These remarks, however, apply to plant for superfine purposes. Castings in these acid-resisting steels are being used on an extensive scale for valves and many other similar purposes.

#### A Few Applications

One of the most important processes in which the austenitic chromium-nickel steel (18 per cent. chromium, 8 per cent. nickel) has been used, and in which, indeed, it has proved invaluable, is the manufacture of nitric acid by the oxidation of ammonia. For absorption towers, acid coolers, and pipe lines using this acid, it is the ideal material, combining strength with very great resistance to corrosion. It is equally useful in dealing with the acid where storage and transport are concerned. In certain manufacturing processes the presence of this acid may confer passivity towards other reagents present which would normally attack the austenitic chromium-nickel steel. In this way it becomes possible to use the austenitic chromium-nickel steel in connection with mixed-acid nitration processes, a matter of great importance in view of the increasing use of nitrocellulose products.

Dyeing is an operation in which more importance usually attaches to the colour of the product than to the effect on the metal, since traces of dissolved metal may have a very great influence on the colour of the dyed fabric, while having little effect on the usefulness of the vessel from the mechanical standpoint. Naturally, with the large number of dyes in use, it is impossible to generalise, but it is satisfactory to note that actual dyeing tests have clearly indicated the general superiority of the austenitic chromium-nickel steel over most

other materials, the colour in many cases being equivalent to that obtained in enamelled vessels. Ease of cleaning gives it a very obvious advantage over wood.

#### Drugs, Chemicals, Foodstuffs, etc.

In the preparation of many drugs and fine chemicals the steel finds another field of usefulness. Here, again, purity of the product is of great importance, and great care must be exercised in the choice of a plant material. For the preparation and storage of foodstuffs, both in the home, hotel, or factory, austenitic chromium-nickel steel is proving itself invaluable. Its complete resistance to the corrosive action of most fruit juices, sauces, pickles, etc., is of the greatest value. Milk and dairy products generally are of very great importance, and it is interesting to note that laboratory work by Hunsicker and others is confirming the opinion of users, that the austenitic chromium-nickel steel is the ideal material for the construction of dairy utensils, storage tanks, etc., not only because of its immunity from attack by the milk and its products, but also because of the ease of cleaning and general corrosion resistance. The need for preventing the lodgment of bacteria makes care in manufacture specially important, the avoidance of crevices, etc., being very important.

Many other uses might be mentioned, such as evaporators for various liquors (*e.g.*, neutral ammonium sulphate solution), pumps and other parts in contact with acid mine waters, fittings exposed to marine conditions or the atmosphere of industrial districts, parts demanding resistance to oxidation at high temperatures and so on.

#### Failures and their Lessons

The use of acid-resisting steels has now attained such dimensions that it is possible to review usefully not only the successful applications, but also a number of instances where trouble has been experienced. Turning to a general consideration of failures which have occurred from time to time with these austenitic nickel-chromium steels, some of these have taken place because the steels have been used for purposes for which they are unsuitable. In such instances it is usually found that trial has been made without consulting the data on corrosion already accumulated. Even in such cases, however, it sometimes happens that unexpected service results. For example, although these steels were not recommended for use with sulphuric acid, it was found that they served admirably for use as trays in sulphuric acid pickling baths. This was due to the fortuitous natural inclusion in the bath of copper sulphate, this product being formed by the action of the sulphuric acid on the nickel silver articles which were being treated. The copper sulphate served as a passivating medium.

Other failures have occurred from purely mechanical reasons, being governed by design and not materially affected by technique of manipulation during production. An example will illustrate this. A cylindrical vessel which failed in service had several appurtenances riveted on. It worked on the vibratory system, and, owing to the fatigue effect, stress cracks occurred from numerous rivet holes. A suitable distribution of a larger number of rivets would have reduced the local stress at the rivets and materially assisted in reducing the liability of breakdown.

The author, however, desires to deal with the other types of failure which can be attributed to manipulation during fabrication or to some treatment given subsequent to fabrication, and which have not been referred to earlier in these notes. These failures may be roughly divided into three categories:— (1) Due to intercrystalline corrosion; (2) Due to intercrystalline corrosion and stress cracks; and (3) Due to stress cracks.

#### Intercrystalline Corrosion

There are not many clear-cut examples of this form of failure, one of the most striking being an impeller casting. This impeller, after being in service for a few months, pumping impure water, was found to be in an advanced state of attack. The primary cause of the trouble was that an unsuitable heat-treatment had been given to the casting to facilitate machining, and a further contributory factor was that the water which was being pumped contained a large percentage of aluminium sulphate, which probably accelerated corrosion owing to hydrolysis.

### Intercrystalline Corrosion Together with Stress Cracking

This is perhaps the most frequent type of failure, and is particularly prone to occur in welded plant. These two features—intercrystalline corrosion and stress cracking—have been coupled together advisedly, because, so far as one's personal experience goes, one cannot call to mind an instance of a welded structure cracking adjacent to the weld during or shortly after welding. If no forces other than mechanical stresses were at work, and given reasonable mechanical properties in the zone adjacent to the weld (and reasonable properties appear to be present when judged by tensile tests on welded samples), one would expect that if fracture were going to occur, it would occur within reasonable time of completing the job. In this connection one calls to mind a particular case of oxidiser cooler tubes, which fractured before being put into service. They had been made some months before, and had been transported to the site without fracture. It was after being kept on the site for some time in the open, immediately adjacent to an acid plant and therefore subject in some degree to an acid atmosphere, that the fractures were noticed. It would, therefore, appear that both corrosion and stress played a part in the failure of these particular tubes, and that softening would have prevented the trouble.

Against this experience there is, of course, the fact that a very large number of similarly welded plants was satisfactory. Variations in welding technique are responsible for the different behaviour, although, so far as can be ascertained, similar methods were adopted in each case.

### Stress Cracking Without Corrosion

Typical instances are those where cold pressing is carried beyond the limit of what the material will stand. Such cracking is intercrystalline, as in the case of certain other metals. All three types of failure are eliminated provided that softening treatment is given previous to service, always assuming that the article or plant does not subsequently attain undesirable ranges of temperature.

## P-M-G Metal

### New Substitute for Gunmetal

P-M-G METAL is an alloy which has recently been produced at the Naval Construction Works of Vickers-Armstrongs, Ltd., at Barrow-in-Furness. The claims made for it by the producers are as follows:—

P-M-G metal is a high-grade copper alloy possessing outstanding qualities. It has a very short freezing range, which approximates from 950° C. to 900° C. and consequently is an excellent casting alloy. It can be readily forged, and in the wrought condition the metal possesses greatly increased strength while retaining ample ductility. It is cheaper to manufacture than gun-metal, and, by its use in castings, porosity and unsoundness troubles are almost entirely eliminated. P-M-G metal is definitely superior to gun-metal in strength and other mechanical properties at normal and elevated temperatures, and has a large range of application where gun-metal, manganese bronze, or other special brasses and bronzes are now in use.

### Professor Hanson's Report

P-M-G metal has been the subject of an investigation by Professor D. Hanson, head of the department of metallurgy at Birmingham University. In his report he states that the P-M-G metal which he has examined has been intended to be used as a substitute for Admiralty gun-metal of the standard composition 88 copper, 10 tin, 2 zinc. P-M-G metal is a copper alloy, but the tin in it is replaced by a special hardener, invented at the works of Vickers-Armstrongs, Ltd. There are no technical difficulties in the production of the hardener, which is inexpensive to manufacture. Professor Hanson remarks that the saving in cost effected by using the hardener instead of tin in Admiralty gun-metal is not less than 15 per cent., and in view of the excellent casting qualities, fewer foundry wasters, and high mechanical properties, substantial additional savings will be obtained.

Professor Hanson's report states that P-M-G metal may be cast into sand or metal moulds, no special precautions being required. It appears to be resistant to oxidation at high temperatures. The sand cast test bars were all very sound, and no defects, pinholes, etc., were revealed during machining.

Professor Hanson was impressed by the soundness of cast P-M-G, all the castings he examined being remarkably free from porosity, a serious trouble in gun-metal. Microscopical examination of a number of specimens cut from castings showed the metal to be exceptionally sound and free from casting defects. The structure was in all cases uniformly fine-grained and free from segregation. P-M-G metal can be readily forged hot.

## Sulphur in Coal and Coke

### Methods of Determination

FROM a metallurgical standpoint the presence of sulphur in the fuels used is of very considerable importance, as in a number of cases contamination of the metals, to a greater or lesser degree, is liable to result. In many metals and alloys, the presence of sulphur in even small quantities has been definitely proved to be harmful, and for this reason a reliable control must be kept on the sulphur content of the fuel used.

### A Works Routine Method

The Eschka method for the determination of sulphur in coal and coke is the one most commonly employed, but this is so well known that it need not be repeated here. A few alternative methods have been proposed from time to time, but the writer has found that the following method is rapid and at the same time sufficiently accurate for works routine control. This method for the estimation of total sulphur is based on the ignition of the sample with sodium peroxide and the determination of the sulphur in the melt as barium sulphate after extraction with water.

Take 0.7 gram of coal or 1 gram of coke and intimately mix it with about 15 grams of sodium peroxide in a 2 in. nickel crucible. Place on this a nickel lid which has a hole pierced through the centre, and place the whole on a piece of aluminium or copper foil in the bottom of a 6 in. porcelain dish which contains about  $\frac{1}{2}$  in. of distilled water. Through the hole in the lid place a piece of platinum or copper wire which has been previously made red hot. This ignites the mixture, and after allowing the crucible to cool, dissolve out the melt in hot water. Transfer to a beaker and make slightly acid with hydrochloric acid (if there is any insoluble matter it must be filtered off). The bulk should now be made up to about 250 c.c., and heated to boiling. While boiling, 25 c.c. of a 10 per cent. solution of barium chloride are added drop by drop. The precipitate is allowed to settle in a warm place, filtered off, ignited and weighed as barium sulphate as usual. A blank determination must be carried out on the materials used so as to avoid any possibility of errors from this source.

### A Recent Suggestion

Another method, claimed to be particularly useful in the case of coals high in sulphur, is proposed by O. Hackel in a recent issue of the *Chemiker-Zeitung*.

One gram of the finely-ground coal or coke, after it has been dried at a temperature of 100° C., is mixed in an iron crucible with eight grams of a mixture of equal parts of sodium carbonate and potassium permanganate, and then the whole mass is covered with another 2 grams of the mixture. The crucible is very carefully heated to redness and maintained at this temperature for about 30 minutes. After cooling, the contents of the crucible are extracted with about 150 c.c. of hot water containing a small quantity of sodium carbonate. This treatment is continued until the mass is freed from sulphate. The filtrate is now made slightly acid with hydrochloric acid and then precipitated with barium chloride solution as usual. In the case of coals or cokes which are high in sulphur, only a  $\frac{1}{2}$  gram sample is taken.

## Cheaper Beryllium and Compounds in Prospect

IN Germany, the firm Siemens and Halske, directors of the Beryllium Research Institute, which was organised in 1923, have developed the basic electrolytic process of Goldschmidt to the point where one ton of the metal can be produced annually. At present it can be sold at 1 gold mark per gram, and with larger demand at a lower price. Up to the present the main uses for compounds of beryllium include the impregnation of gas mantles with the nitrate and as a laboratory reagent for certain other salts.

## Metallurgical Topics: Monthly Notes and Comments

### From Our Own Correspondents

#### A Book on Tin

IN the past ten years or so, the American Chemical Society has proceeded steadily and efficiently with its task of the production and publication of scientific and technical monographs on chemical and allied subjects. The latest of the series (known as the American Chemical Society Monograph Series) is *Tin: Its Mining, Production, Technology and Applications*, by Dr. C. L. Mantell, of the Pratt Institute, Brooklyn (New York: The Chemical Catalog Co., pp. 366, \$7).

The author has covered the subject of tin in a somewhat comprehensive manner, dealing with the history, physical and chemical properties, production, distribution and consumption of the metal. The ores and ore deposits, the ore dressing, mining, smelting, refining and metallurgy represent the distinctly metallurgical sections, and are treated somewhat from the point of view of the metallurgical chemist. The industrial applications of tin in its simpler forms, as in plating, hot-dipped coatings, foil and collapsible tubes, are dealt with rather from the chemical than the metallurgical engineering point of view. The section on alloys is built up on the basis of physical metallurgy, and the sections on corrosion from the point of view of the chemical engineer.

The chapter headings are as follows:—History; Physical and Chemical Properties; Production, Distribution and Consumption; Ores and Ore deposits; Mining and Ore-Dressing; Smelting and Metallurgy; Gaseous Reduction; Electrolytic Refining; Plating; Alloys; Hot-Dipped Coatings; Foil and Collapsible Tubes; Compounds; Corrosion; Corrosion of Tin Plate by Food Products; Secondary Tin; Detinning of Tin Plate Scrap; Analytical Methods.

#### B.E.S.A. Specification for Phosphor Bronze

A SPECIFICATION for phosphor bronze bars or rods has recently been issued by the British Engineering Standards Association. This specification is drawn up on parallel lines to the specifications for brass bars published in 1926, and covers round, square, and rectangular bars from  $\frac{1}{4}$  in. to 2 in., and hexagon bars for standard nuts from No. 3 B.A. to  $1\frac{1}{4}$  in. B.S.W. and  $1\frac{1}{8}$  in. B.S.F., with specified margins of manufacture. The chemical composition of the material, mechanical tests, and the procedure of selecting samples for these tests are laid down. Copies of the Specification, No. 369-1929, can be obtained from the British Engineering Standards Association, Publications Department, 28, Victoria Street, London, S.W.1, price 2s. 2d., post free.

#### Atmospheric Corrosion of Metals

THE Faraday Society has just published "The Third (Experimental) Report to the Atmospheric Corrosion Research Committee of the British Non-Ferrous Metals Research Association," by Dr. J. C. Hudson (pp. 380, 5s. 6d.), the volume also including the discussion on the report which was held by the Faraday Society on May 23. This is the third report on the results of an investigation on the atmospheric corrosion of non-ferrous metals and alloys which has been in progress since 1920. The first two were presented by Dr. W. H. J. Vernon, and, in presenting the third, Dr. Hudson points out that it differs from its predecessors in that it deals exclusively with field tests in the open, whereas the others were more particularly concerned with the results of exposure tests in several types of indoor atmosphere.

Sixteen materials were tested, including copper, nickel, zinc, lead, and some of their most important alloys. In order to obtain information on the effect of local conditions, tests were carried out at five different stations. The report is divided into the following sections:—Organisation of field tests; tests by the weight-increment method (Stevenson screen tests); tests by the electrical resistance method (complete exposure); loss in weight tests; and general discussion of the results.

#### Complex Copper-Aluminium Alloys

THE current issue of the *Revue de Métallurgie* (No. 9, 1929, pp. 464-487) contains an account of an investigation by Ernest Morlet on copper-aluminium alloys containing manganese, tin and cobalt. The first portion deals with copper-

aluminium-manganese alloys, for the preparation of which pure electrolytic copper, commercial (98.25 per cent.) aluminium and cupro-manganese (Cu, 69.9; Mn, 28.2) were employed, the method of making the alloys being described. They ran from 83.32 per cent. of copper to 89.07; 6.58 per cent. of aluminium to 10.34; and from 1.05 to 5.92 per cent. of manganese. The iron associated both with the aluminium and with the cupro-manganese used varied from 0.55 per cent. to 0.94 per cent., from which it will be seen that the alloys, although described as ternary, were really quaternary metals. Cooling and heating curves showing the transformations they underwent are given in the paper. It was found that the resistivity of the series of alloys prepared increased with the percentage of manganese present; that the e.m.f. fell and the hardness rose in the same way; but the hardness varied, in some cases actually fell, as the structure of the alloys altered on heat treatment. In some of the alloys, quenched at 850° C., a distinctly martensitic structure was observed. The investigations on the copper-aluminium-tin, and the copper-aluminium-cobalt alloys will be described, and the research summarised as a whole in succeeding issues of the *Revue de Métallurgie*.

#### Steel in Building Construction

THE Institution of Structural Engineers is noted for the high standard of papers read before its members, and one read at a recent meeting, by Mr. G. S. Bowers, which was well up to that standard, and was entitled "Some American Comparisons"—not all by any means in favour of British practice—naturally evoked a lively discussion. He claimed, among other things, that American engineers have a better and wider range of structural steel sections to choose from in building construction, and that practice in these respects in Great Britain is hemmed in by hide-bound authorities, regulations and old-established practices. The need of cheapening building construction in steel is, of course, fully recognised, and, indeed, a new body, the British Steelworks Association, has just come into being to extend by all means in its power the use of British steel, which can, of course, be done only by this same method of lowering costs. Mr. Bowers advocates the reduction of live loads, and lighter external wall construction, as well as increased permissible working stress on steel, which, he suggests, should be 9 tons per sq. in. He had much to say about L.C.C. regulations, which he seems to regard as antiquated and a hindrance to progress. In other parts of the world, buildings are being constructed up to 800 ft. in height; here at home we remain at round about 100 ft. Technically, he seems to say, a great deal more steel could be used, and to much better advantage. At a time when the steel industries are depressed and looking for work, Mr. Bowers's remarks seem to need serious consideration.

#### Hindrances to Progress

IT is not only the engineer's job, but also the metallurgist's job, to see that all the steel that can be used is used, and to the best advantage. If our regulations are too strict it devolves on the metallurgist to prove that less of his metal will adequately discharge the task it is called upon to fulfil; wasteful use does no one any good in the long run. And he must convince the engineer. Between them they can surely bring recalcitrant authorities to book, seeing that the authorities decree as the engineer advises them to.

There are many things done in iron and steel abroad that are not done here, and that, if done, would stimulate the use of steel in building construction. One of the things so done was not, however, even mentioned in Mr. Bowers's paper. This is the use of welding in building construction, in respect of which great advances have been made of late, with much success, in America as well as on the Continent, especially in Germany. It would appear that in regard to welding the L.C.C. regulations again block the way. The saving to be effected by welding structures instead of riveting is said to extend far beyond the actual amount of steel that need be used; labour and other costs fall in proportion. The structures are not weakened by the rivet holes, and welding provides as staunch and sound joints as can be made by any other method. The smaller amount of steel used per building

is more than compensated for by the stimulus offered to building operations. The whole question of welded steel construction calls for close study and development.

### Gas Producers for Metallurgical Furnaces

OF the many excellent papers read at the recent meeting of the Institute of Fuel, that by Mr. J. S. Atkinson on "The Installation and Operation of Gas Producers for Metallurgical Furnaces" is probably the most interesting to metallurgists. Electric furnaces, both for heating and annealing, have many attractions, but it is almost time that gas heating came in for some share of attention, as it has many features to commend it, not the least being its cheapness. The paper is, however, restricted to a description of the merits, chiefly, of the Stein-Chapman producer and its latest modifications, and hardly does justice to the larger subject of gas-producer practice in metallurgy generally. The author, however, brings out usefully the need of designing metallurgical furnaces in strict correspondence with the type of gas they are intended to use, a precaution which will prevent much disappointment and expense amongst the school of thought which appears to imagine that all gases behave alike, and that any kind of gas will do in any kind of furnace. For an ideal producer gas, moreover, the fuel needs careful selection; here again it is a mistake to expect the best results with indifferent coal. The physical characters of the latter are equal in importance to the chemical composition, the nature of the ash and its fusion temperature being decisive factors in the behaviour of a producer. The types mentioned in the paper are described in detail and well illustrated, and the paper as a whole is a useful addition to a literature which is voluminous but not, as a rule, very informative.

### Protective Coatings for Aluminium

PROTECTIVE coatings for aluminium and its alloys become increasingly important as the progress of aeronautics conquers new fields. The paper read by Mr. Sutton at the last meeting of the Electroplaters' and Depositors' Technical Society was timely, and contained a good deal of useful information within a small compass. The anodic oxidation process was given pride of place, and the method of securing an effective coating by this means was described in some detail.

Mr. Sutton advocated the use of steel tanks as containers for the chromic acid electrolyte, the necessity of keeping them clean and free from rust being emphasised. Graphite plates were recommended as cathodes. Care must be taken to avoid the presence of chlorine and of chlorides, and the sulphate content should likewise be kept as low as possible. Other processes described were the Jirotko process, popular on the Continent and capable of development from the decorative point of view; the Stafford O'Brien process, said to be one of anodic treatment at low voltage in sulphuric acid containing a chloride; and the "Alclad" method, whereby duralumin is coated with pure aluminium. It is claimed, in respect of this method, that in the case of cut edges the coating suffers by proxy, instead of the duralumin itself. Finally the author dealt with electro-deposits on aluminium, instancing in this connection nickel, zinc and cadmium. Zinc, with proper precautions, is said to afford better protection to aluminium, under severe conditions, than nickel, while cadmium plating on aluminium alloys is said to give quite good results.

### The Rare Metals

PUBLICATIONS of the Bureau of Mines, Department of Commerce, Washington, and especially those which deal with the mineral resources of the United States, are amongst the most valuable publications to which metallurgists can refer. No. 1: 17, on "Rare Metals," although comprising 60 pages, is a *vade mecum* on its subject, for it is not only statistical, but gives abstracts—and very good abstracts, too—of the chief sources of information. The metals dealt with are, however, hardly what are understood in this country as "rare metals," as they include nickel and cobalt, as well as some comparatively rare metals such as molybdenum, tantalum, titanium, tungsten and vanadium, and finally, the incontestably rare metals, radium and uranium. Cobalt is chiefly derived from that modern El Dorado, Katanga, and from Queensland and Canada, and the annual production may be estimated at somewhere about 1,000 tons. It has nothing like the decolorising effect on copper and gold that nickel has. The world's output of molybdenum is of the order of 3,000 tons

per annum. The United States, Mexico and Norway are the chief producers. The uses of tantalum are described as so few that an unforeseen addition to world supplies would be unlikely to increase demand, but only to reduce prices. A detailed description of the Western Australian deposits is given. The account of radium is hardly up to date; those available through our own Department of Industrial and Scientific Research contain later and fuller details. In the case of each of the metals a valuable bibliography is appended.

### The Steel Fusion

IT was recently announced that a provisional agreement had been reached between Dorman, Long and Co. and Bolckow, Vaughan and Co. for a fusion of interests by means of an exchange of shares. The capital obligations of these two undertakings, owning iron, steel, and colliery properties, principally in Durham and Yorkshire, amount to nearly £20,000,000.

When the scheme now submitted has been sanctioned, Dorman, Long and Co., which will take over the assets of Bolckow, Vaughan and Co., will raise its loan and share capital from £12,253,756 to £17,103,919. This will be divided as to £5,855,773 in debenture stock and as to £11,248,146 in preference and ordinary shares. Although the combined undertaking will be known as Dorman, Long and Co., for some time it is proposed to include in the title "Incorporating Bolckow, Vaughan and Co."

Negotiations have been taking their course for some considerable time, and it is now stated that the respective boards, convinced of the advantages of amalgamation, instructed Sir William McLintock to prepare a scheme, which has been approved by Sir William B. Peat and Sir William Plender, acting for Dorman, Long and Bolckow, Vaughan respectively.

### Scope of the Fusion

IN the statement announcing the fusion it was pointed out that "Dorman Long and Co. and Bolckow, Vaughan and Co. are parallel with each other in equipment and control of the raw materials of steel production and in the market they supply. Amalgamation between them offers important advantages, in addition to the general benefits to be derived from consolidation. Their joint power of production will be based upon a well-balanced supply of raw materials and a more efficient use of plant. Concentration of works in the most economical units, with a consequent reduction in costs, will enhance the earning powers of the combined concern. Expenditure on future developments will result in greater efficiency and economy, since it will be undertaken by a single company and not by two competing units."

Bolckow, Vaughan control Redpath, Brown and Co., own half the share capital in Darlington Rolling Mills, and, jointly with Cortonwood Collieries, have a controlling interest in the Upton Colliery Co. Dorman, Long and Co., manufacturers of constructional steel, absorbed Bell Bros., Sir B. Samuelson and Co., Carlton Iron and Steel, etc., and own coal, ironstone mines, blast furnaces and coke ovens in Durham and Yorkshire. They control, among other companies, Pearson and Dorman Long, Ltd.

### The European Steel Cartel

INFORMATION on the results of the discussion at the recent Vienna meeting of the steel cartel is still incomplete because those who are in charge of the negotiations for the renewal of the pact are maintaining a rigid discretion, and this fact is interpreted to mean that there are yet serious difficulties to be surmounted. On the question of renewing the cartel it is now known that an understanding could not be reached because the schemes put forward were so many and various. The German internal understandings, which expire in December and in the spring of next year, had to be taken into account, for in the circumstances the German group of steel producers could not take a decision in the matter of prolonging the cartel for a certain number of years.

All that could be done was to prolong the pact until April next year, and in the meantime the German internal negotiations can proceed, and it will be known if the reconstituted German organisations will be able to take part in the definite negotiations for a renewal of the pact. It is known, however, that each group adhering to the cartel presented a number of claims. The quota of German exports has not been interfered with; Germany can export a maximum of 300,000 tons monthly, but this figure has been increased as compensation for lock-out losses.

## Some Inventions of the Month

### By Our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature, published weekly in THE CHEMICAL AGE.

#### Alloys

**COPPER-ALUMINIUM** alloys of high casting capacity and low degree of shrinkage contain 87-93 per cent. of copper, 3-9 per cent. of aluminium, 1.25-2.5 per cent. of silicon, and 1-3 per cent. of iron. The addition of manganese in amounts up to 0.2 per cent. is advantageous. Preferably, the copper, aluminium and silicon are melted together by the usual methods, and the iron is introduced into the melt in the form of cast iron powder or iron-silicon or iron-manganese alloys. The incorporation of the iron or of the iron alloy with the other elements to form the complete alloy is facilitated by the presence of fluorides, such as sodium silicon fluoride or sodium aluminium fluoride, and aluminium powder. See Specification 318,802, dated November 29, 1928, by A. Pacz, of Cleveland, Ohio, U.S.A.

#### Copper

IN a process for refining copper described in Patent Application 317,399, bearing the International Convention date, August 15, 1928, by H. H. Stout, of Douglas, Arizona, U.S.A., the molten metal is oxidised by air or oxygen in a preheated furnace at about 1090-1120° C., reduced by poling, charcoal, or reducing gases in the same or another preheated furnace at about 1220-1245° C., and cast without further contact with combustion gases.

#### Iron

A PROCESS for effecting the concentration of iron in ores or other materials containing it is described and claimed in Specification 319,309 (Dicker), a communication from Bradley-Fitch Co., of Minneapolis, Minnesota, U.S.A., dated June 19, 1928. The materials are subjected to the action of a reducing gas at a relatively low temperature of the order of 400-500° C., so as to render more of the iron content susceptible to magnetic concentration, whereafter they are subjected to magnetic concentration. The presence of water vapour during the heat treatment is found to be advantageous in most cases, and such water vapour is preferably derived from a partial combustion of the reducing gas, containing free or combined hydrogen, with air or oxygen, this partial combustion serving also to supply the heat for attaining the desired temperature. The process is particularly applicable to limonite and hematite ores.

IN refining iron, steel, or alloys thereof, the last trace of oxygen is removed by adding to the molten metal an alloy of lead and sodium, or a lead cartridge filled with sodium. Alternatively, the sodium may be replaced by any other suitable deoxidising agent, and the lead by any metal which is of higher specific gravity than iron and is also not detrimental to the iron alloys. See Patent Application 317,493, bearing the International Convention date, August 18, 1928, by A. Glazunov, of Pribram, Czecho-Slovakia.

**MOLTEN** iron is refined in a converter or open-hearth furnace by means of water introduced into the bath in a liquid or gaseous state. The water is fed in at such a temperature, and at such a distance from the bath, that part of it is decomposed, whereupon the oxygen exerts a fining action on the iron, and the hydrogen agitates the bath. The gases rising from the bath are burnt by means of a supply of cold or preheated air. See Patent Application 317,373, bearing the International Convention date, August 14, 1928, by F. Krupp Akt.-Ges. Friedrich-Alfred-Hütte, of Rheinhausen, Germany.

#### Pure Iron

A METHOD of producing pure iron is described in Specification 318,499 (Johnson), dated April 2, 1928, a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany, of a Patent of Addition based on Specification 269,345 (see THE CHEMICAL AGE, Vol. XVII, p. 7 [Metallurgical Section]). According to the method of the parent specification, iron containing carbon and oxygen produced by the decomposition of iron carbonyl was melted, or merely heated to sintering, out of contact with air, and the main difficulty was that of ensuring that the ratio of carbon to oxygen present was such as to effect their complete elimination as oxides of carbon. According to the present improvements, iron obtained from iron carbonyl and containing an excess of

carbon over the amount necessary for binding all oxygen present, is mixed with iron obtained from iron carbonyl and containing an excess of oxygen over the amount necessary for binding all carbon present, the proportions of the mixture being such that the carbon and oxygen are eliminated completely as carbon oxides on melting or sintering.

#### Wrought Iron

**IMPROVEMENTS** in the manufacture of wrought iron by the processes of Specifications 170,124 and 224,941 (see THE CHEMICAL AGE, Vol. XII, p. 7 [Metallurgical Section]) are described in the series of Patent Applications 315,826-9, bearing the International Convention date, July 19, 1928, and 315,858, bearing the International Convention date, July 21, 1928, by A. M. Byers Co., of Pittsburg, U.S.A. The molten product of a steel-making operation is poured into molten slag having the necessary puddling characteristics, and the temperature of the slag is maintained sufficiently below the melting point of the metal during the pouring operation to cause the product to be granulated as it enters the slag bath. The improvements are as follows:—

315,826. The molten slag remaining in the receptacle after the granulation has taken place is poured into another receptacle, and cold additions such as iron oxides are made to it before pouring in more of the molten metal, whereupon the surplus slag is removed and treated as before.

315,827. The pouring ladle for the metal is moved to and fro over the slag bath or provided with a plurality of spouts to obtain a better distribution over the surface of the slag.

315,828. The steel from a Bessemer converter is poured in one or more streams from a height of not less than 6 feet above the slag level into a slag bath 3-4 feet deep, at a rate of about  $\frac{1}{2}$  ton per minute in each stream.

315,829. The metal is bessemerised before pouring it until the carbon content is below 0.10 per cent., the combined silicon and carbon content less than 0.2 per cent., and the oxygen content less than 0.31 per cent.; the slag used has a silica content of not over 15 per cent.

315,858. The slag used is prepared by melting slag-forming materials such as iron ore, mill-cinder, etc., and silicates such as gravel, in a non-reducing atmosphere in a hearth furnace.

#### Manganese

IN Specification 319,392 (Dicker), a communication from Bradley-Fitch Co., of Minneapolis, Minnesota, U.S.A., dated June 19, 1928, a process is described for recovering the manganese values from liquors obtained by leaching, with solutions of ammonium salts, products obtained from manganese-iron ores, as described in Specification 284,098 (see THE CHEMICAL AGE, Vol. XVIII, p. 182), which products contain the manganese in the form of manganous oxide. An oxidising gas, such as air or oxygen, and a limited amount of ammonia, are used to effect the selective precipitation of iron from the liquor without substantial precipitation of the manganese values. Ammonia may be supplied to the liquor directly or may be generated by addition of caustic alkali, but it is preferred to generate it by addition of reduced ore containing manganous oxide. A diagram is given showing the complete arrangement of plant.

#### Metals, etc.

**METALS** (especially those of the chromium and titanium groups) or metal compounds are heated, preferably in powdered form, in a hydrocarbon-containing atmosphere, at a temperature below 2500° C., and preferably between 1400° and 1600° C., to obtain metals or alloys containing hydrocarbons. A preliminary heating to 900-1000° C. in a hydrocarbon-containing atmosphere may be employed. The treatment may be effected under pressure and in presence of a catalyst, and is applicable also to the surface-hardening of metal articles. See Patent Application 316,103, bearing the International Convention date July 20, 1928, by Bayerische Metallwerke Akt.-Ges., of Munich, Germany.

#### Tin

THE recovery of tin from ores, slags, or tailings containing it is described in Patent Application 316,177, bearing the International Convention date, July 24, 1928, by Mitsubishi Kogyo Kabushiki Kaisha, of Tokio, Japan. The ores, etc., are mixed with a sulphide, sulphate, or sulphite and a reducing agent, and heated, e.g., in a rotary kiln, to 1,000-1,100° C., in a current or atmosphere of non-oxidising gas, whereby the tin is converted into and volatilised as sulphide.

## Current Articles Worth Noting

We give below a brief index to current articles in the technical Press dealing with metallurgical subjects.

**ALLOYS.**—The limit of solubility of copper in the reversible ferro-nickels. P. Chevenard. *Comptes Rendus*, October 14, pp. 576-578 (in French).

The electrolytic preparation of alloys of barium and strontium. K. W. Ray. *Metals and Alloys*, September, pp. 112-113.

**ANALYSIS.**—The determination of small amounts of nickel in steel. B. Jones. *Analyst*, October, pp. 582-589.

**BERYLLIUM.**—Beryllium. M. Merlub-Sobel. *Metals and Alloys*, August, pp. 69-70.

**CORROSION.**—The corrosion of aluminium alloys in superheated steam. L. Guillet and Ballay. *Comptes Rendus*, October 14, pp. 551-553 (in French).

The attack of ammoniacal solutions on aluminium. J. Calvet. *Comptes Rendus*, September 30, pp. 485-486 (in French).

The effect of the acid anion on the velocity of solution of aluminium. M. Centnerszwer. *Zeitschrift Elektrochem.*, September, pp. 695-701 (in German).

Comparative investigations on the resistance to solution and the action of rust on iron tubes protected at the surface. W. Beck and K. Jacobsohn. *Korrosion und Metallschutz*, September, pp. 202-207 (in German).

**ELECTRODEPOSITION.**—The phenomena occurring in the separation of chromium from solutions of chromic acid. E. Liebreich and V. Duffek. *Berichte*, October 9, pp. 2527-2538 (in German). Evidence is shown for the assumption that deposition occurs by discharge of the divalent (chromous) ion of chromium.

The electrodeposition of chromium from aqueous solutions of chromic acid. J. Roudnick. *Bulletin Soc. Chim. Belgique*, August-September, pp. 276-294 (in French).

**GENERAL.**—Volume changes during the solidification of metals and alloys of low melting-point. W. E. Goodrich. *Transactions Faraday Soc.*, October, pp. 531-569. The following metals were investigated: tin, lead, bismuth and zinc; and the following alloys: lead-tin, bismuth-tin, antimony-tin (0-15 per cent. of antimony), lead-antimony (0-25 per cent. of antimony), tin-antimony-copper, tin-lead-antimony, zinc-copper-tin, and zinc-copper-aluminium.

The thermochemistry of iron, manganese and nickel. W. A. Roth. *Zeitschrift angewandte Chem.*, October 12, pp. 981-984 (in German). The heats of a series of oxidation and reduction processes involving manganese, iron and nickel have been redetermined.

The electrochemical behaviour of gold and silver in hydrochloric acid solution. G. Grube. *Zeitschrift Elektrochem.*, September, pp. 703-709 (in German).

Progress on the problem of transverse fissures in rails. M. E. McDonnell. *Metals and Alloys*, September, pp. 93-97.

The distribution equilibrium of silver between lead and aluminium: A contribution to the testing of the distribution law for condensed systems. R. Lorenz and F. Erbe. *Zeitschrift anorganische Chem.*, Vol. 183, Part 3, pp. 311-339 (in German).

Radiography as a tool in the metal industry. W. L. Fink and R. S. Archer. *Transactions Amer. Soc. Steel Treating*, October, pp. 551-599. This paper gives typical examples of the applications of X-radiography in the laboratory of the United States Aluminium Co., where it has been in regular and successful commercial operation for two years.

Improving of refractory lining of heat treating furnaces for high temperature annealing of steel castings. W. J. Merten. *Transactions Amer. Soc. Steel Treating*, October, pp. 600-606.

Economical re-use of solid carburising materials. H. B. Knowlton. *Transactions Amer. Soc. Steel Treating*, October, pp. 606-625.

**IRON.**—The solubility of oxygen in solid iron. W. Krings and J. Kempkens. *Zeitschrift anorganische Chem.*, Vol. 183, Part 3, pp. 225-250 (in German).

Comparative properties of wrought iron made by hand-puddling and by the "Aston" process. H. S. Rawdon and O. A. Knight. *Metals and Alloys*, August, pp. 47-56. The new ("Aston") process differs radically from the conventional hand-puddling process in that the pig-iron, after being melted in a cupola, is refined in a Bessemer converter and then incorporated with the slag, which is prepared in a separate furnace.

**MECHANICAL PROPERTIES.**—A study of the Ikeda accelerated test for determining endurance limit. H. F. Moore and S. Konzo. *Metals and Alloys*, August, p. 70.

**STEEL, ALLOY.**—The characteristics of V2A-steel. E. Liebreich. *Korrosion und Metallschutz*, September, pp. 199-202 (in German).

**STEEL, HEAT-RESISTING.**—The use of steel in the oil industry, with a special reference to heat resisting steels. Sir R. Hadfield and S. A. Main. *Journal Inst. Petroleum Tech.*, October, pp. 611-633.

## Chemically Pure Iron

MR. T. D. YENSEN, in charge of the magnetic section at the works of the Westinghouse Electric and Manufacturing Co., of East Pittsburgh, writes as follows:—"The reference in the October 5 issue of your periodical to my paper, 'On the Road to Pure Iron,' is much appreciated by the author. The statements are clear and substantially correct with one exception, and, as this is an important point that may readily lead to confusion, I would suggest that you have it corrected in your columns. You say: 'With 0.05 to 0.06 per cent. carbon present permeabilities up to 60,000 have been obtained.' This should obviously be: 'With 0.005 to 0.006 per cent. carbon present . . . With 0.05 to 0.06 per cent. carbon, the permeability would be less than 5,000.' The point in question was made in a paragraph in the Monthly Metallurgical Section of October 5, p. 27, entitled 'Elimination of Carbon and Oxygen.'

## Commercial Intelligence

The following are taken from printed reports, but we cannot be responsible for any errors that may occur.

### Mortgages and Charges

[NOTE.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debts due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case, the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.]

BEARDMORE (WILLIAM) AND CO., LTD., London, E.C. Registered October 14, £5,830 debenture, to Eastern Smelting Co., Ltd., Stafford House, King William Street, E.C.; charged on properties in the Kingdom of Siam, etc., also general charge. \*£1,616,940. November 8, 1928.

EASTERN SIAM TIN DREDGING, LTD., London, E.C. Registered October 14, £5,830 debenture, to Eastern Smelting Co., Ltd., Stafford House, King William Street, E.C.; charged on properties in the Kingdom of Siam, etc., also general charge. \*£5,000. October 25, 1928.

LAFFLY (ENGLAND) CO., LTD., London, N.W., iron founders. Registered September 24, £610 debenture, to A. B. Staples, 33, Old Change, E.C., silk merchant; general charge (including 20, 22, 24, 26 and 35, Lonsdale Road, Willesden). \*—, March 7, 1928.

MERU TIN, LTD., London, E.C. Registered October 14, £5,000 debenture, to Eastern Smelting Co., Ltd., Stafford House, King William Street, E.C.; charge on property at Meru, Ipoh, F.M.S., also general charge.

### Satisfaction

BWANA M'KUBWA COPPER MINING CO., LTD., London, E.C. Satisfaction registered September 28, £10,675, part of amount registered April 13, 1928.





## Monthly Metallurgical Section

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**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

### Protecting Aluminium and its Alloys from Corrosion

#### Paper and Discussion at Electroplaters' and Depositors' Society

The first meeting of the 1929-30 session of the Electroplaters' and Depositors' Technical Society, which took place recently, was devoted to a discussion on a paper read by Mr. H. Sutton, of the Royal Aircraft Establishment. This was entitled "Some Recent Developments in Protective Coatings of Aluminium and its Alloys," and dealt in an interesting way with the latest developments in the production of protective coatings on metals, more particularly of those methods of which the author had had personal experience.

THE anodic oxidation process due to Bengough and his collaborators, said Mr. Sutton in his paper, has been described in several papers, and in a publication of the Department of Scientific and Industrial Research. The process consists in making the article to be treated the anode in a bath of 3 per cent. aqueous chromic acid solution, the voltage being raised gradually from zero to 40 volts in 15 minutes, maintained at 40 volts for 35 minutes, raised to 50 volts in the course of five minutes, and maintained at 50 volts for a further five minutes. By this means a protective coating of oxide is produced on all parts of the surface accessible to electrolyte.

Graphite plates have been used extensively as cathodes, and in general have given satisfactory service. It has been found possible to use clean steel plates as cathodes, or, where convenient, the tank itself, but stainless steel is preferable to ordinary steel for the purpose. A steel containing 0.10 to 0.15 per cent. of carbon and 12 to 14 per cent. of chromium has given the best results as a material for cathodes and tanks. The steel is not an easy one to weld, but, by the use of special technique, can be worked up into satisfactory tanks for anodic plant. In the operation of the anodic process there is a tendency for the chromic acid to be reduced at the cathodes and thereby rendered useless. The amount of reduction occurring is influenced greatly by the nature of the cathodes; for example, stainless steel of the type mentioned has been found to cause less reduction than any other material so far employed.

Chlorine and chlorides are very undesirable impurities and have given rise to considerable trouble, due to the fact that they cause breakdown of the anodic film and severe local attack on the aluminium or alloy during treatment in the normal manner. The chloride content of the electrolyte should not be more than the equivalent of 0.02 gram of NaCl per 100 cc., in order to avoid trouble from this cause.

#### Applications of Anodic Process

The anodic process can be applied to aluminium, duralumin and most aluminium alloys, but alloys containing more than 5 per cent. of copper have not proved capable of being treated. Duralumin, Y alloy and similar alloys can be treated equally well, whether in the final heat-treated and aged condition or after softening treatment. Provided that reasonable care be taken to avoid scratching the film, etc., pieces can be heat-treated satisfactorily after anodic treatment, but if a salt bath be used the film is likely to be injured to some extent unless the salt, usually sodium nitrate, is of fairly high purity.

There is a marked difference between the appearance of softened duralumin and of heat-treated aged material after anodic treatment, the former being rendered a dark grey compared with the latter. This difference has occasionally proved of service as a method of distinction. Attempts to treat unsuitable alloys are very injurious to the bath, since pitting and solution of the aluminium occur. Contact of aluminium or alloy under treatment with steels prevents normal operation of the process; contact with brass results in rapid and severe attack on the brass during treatment. The anodic oxidation process tends to open up and reveal clearly

any defects such as laps, seams, etc., in the metal occurring at the surface; also to disclose any attempts to plug up blow holes, holes drilled in the wrong place, etc.

The protection against corrosion afforded by anodic films is much improved by the subsequent application of suitable paints or enamel, or by a grease. Lanolin has been much used for the purpose, and is probably applied most conveniently in the form of a solution consisting of white spirit 45 per cent., solvent naphtha 45 per cent., and lanolin 10 per cent. by volume.

#### The Jirotko and Stafford O'Brien Processes

The Jirotko process consists in producing a protective film on the surface of aluminium and certain alloys by immersion in a bath consisting of an aqueous solution of suitable salts, notably chromates and salts of the heavy metals, kept at a moderate temperature. The treatment affords considerable protection against corrosion. A very interesting aspect of this process is the variation in colour of the films obtainable, suggesting considerable scope for the process in decorative work.

Precise details are not available of the Stafford O'Brien process, but it is stated to consist of anodic treatment at low voltage in sulphuric acid containing a chloride, and is a method of producing films on aluminium and aluminium alloys for protection against corrosion. It is claimed that the process is effective on aluminium alloys, including the high-copper alloys.

#### Coatings of Metallic Aluminium

A form of duralumin having a coating of aluminium of high purity, and known as Alclad, is being produced, the object of the coating of pure aluminium being to protect the underlying duralumin from corrosion. The coating is understood to be applied by means of a special casting process, and not to the sheet as such. The makers claim that the attack by a corroding agent at cut edges is borne by the metal of the coating, and not by the duralumin.

The protective effect of treatments of the "calorising" type on steel is well known. "Calorising" consists in packing the pieces in a mixture of aluminium powder, aluminium oxide, etc., and heating for a period of some hours. As a result of the treatment aluminium is absorbed by the steel near the surface, and the aluminium-rich layer at the surface has the effect of rendering the steel particularly resistant to oxidation when heated in air.

Another way of applying aluminium to steel is to electro-deposit a thin coating of cadmium on the steel, which is then preheated slightly and dipped in a bath of molten aluminium. The cadmium coating presumably melts and vaporises, permitting access of the molten aluminium to the steel in the absence of air. When the steel article is removed from the bath it has a coating of aluminium resembling that of tin or zinc on work removed from hot-tinning or hot-galvanising baths. The aluminium-coated iron is very resistant to oxidation at elevated temperatures, and to corrosion at normal temperatures, as mentioned by Evans.

Nickel deposits on aluminium have proved capable of affording a fair degree of protection against corrosion, which is, however, dependent upon the physical character of the deposit, since any discontinuities in the coating form seats of corrosion of the underlying aluminium. Brook and Stott observed moderate resistance to corrosion of aluminium samples having nickel electrodeposits of about 0.0001 in. to 0.001 in. thickness. H. K. Work has described methods of producing a roughened surface on aluminium and aluminium alloys by the use of etching solutions. His objection to sand-blasting is the equipment required. His nickel plated samples bearing 0.0005 in. of nickel alone appear to have given fair results in a salt solution test—i.e., partial immersion in 1 per cent. NaCl and 1 per cent. CaCl<sub>2</sub> solutions.

Cournot and Bary made corrosion tests by partial immersion in an artificial sea water, and nickel deposits on aluminium gave fairly good results. It is generally agreed that for reasonable protection of aluminium and aluminium alloys, nickel deposits require to be free from pores and of moderate thickness. Practical experience of duralumin and aluminium plated with nickel has shown that unless the nickel deposit is moderately thick, i.e., 0.0005 inch or more, and substantially free from pin-holes, the deposit may be expected to blister and to become disrupted in places.

#### **Zinc and Cadmium Deposits**

Zinc deposits on aluminium and aluminium alloys have given promising results, and afford better protection than nickel deposits under severe conditions. In the author's laboratory sand-blasting with fine sand has been found the most consistent and generally satisfactory method of preparing the work. Deposits from sulphate and cyanide zinc baths are being investigated for general and protective properties. At present the sulphate type of bath appears to be preferable to the cyanide type, since a higher degree of protection is obtainable by the former, when used with appropriate addition agents.

Cadmium deposits on aluminium and aluminium alloys have given promising results. Cournot considered the cyanide type of bath the best for the purpose, and obtained satisfactory results in corrosion tests. He has also reported favourably on deposits of chromium on cadmium as a protection against corrosion. In the author's laboratory, cadmium deposits have given good results, particularly on aluminium alloys. Although, under marine conditions, the cadmium of the coating appears to be removed rather quickly, it appears to leave a protective coating, probably consisting of mixed oxides of cadmium and aluminium.

Research at the Royal Aircraft establishment on the use of electrodeposited coatings for the protection of metals against corrosion is now receiving assistance from the Department of Scientific and Industrial Research, and special investigations are in hand for the Electrodeposition Committee of the Department.

#### **Discussion**

The chairman (Mr. U. R. Evans) said that the properties of the light alloys were bound up with the invisible surface film which Mr. Sutton had been the first to isolate, and the study of this film was therefore of great importance. Attempts to plate upon aluminium and its alloys formed an interesting study, since the difficulties in plating upon other metals were magnified or "caricatured," resulting in valuable information. With regard to coating steel with aluminium to prevent corrosion, results had been most promising in a comparison which he had carried out between the efficiency of various finishes. There was a big future for aluminium coatings if their performance was satisfactory and costs not abnormally high.

Mr. E. A. Ollard said he was interested to hear from the chairman that Mr. Sutton had been the first to isolate the oxide film. Actually, when he was in Berlin in 1924, Dr. Leibreich had shown him a photograph of an aluminium electrode taken at the moment of the reversal of the current: this clearly showed the oxide film curling away from the surface. He thought Mr. Sutton's notes on plant used in the anodic process would be particularly interesting to the practical chromium plater, who met similar difficulties. In regard to depositing upon aluminium, Professor Desch had obtained good deposits of cadmium, using a sulphate solution. Did the same apply to zinc?

Dr. R. S. Hutton, the president, said although the idea of

anodic oxidation was not new (the aluminium rectifier had been known some 20 years ago), yet the idea had been lying dormant until Bengough and his colleagues at the Air Ministry had worked upon it and brought it to the point of commercial application. There was a very big potential field for the process, and licences to work the process were now being issued to manufacturers (chiefly in the aircraft industry) by the Department for Scientific and Industrial Research. There was also a big future for aluminium coatings, although an electrodeposit which was much thinner might give as good and even superior results.

Mr. D. J. MacNaughtan, referring to the practical difficulties in anodic oxidation, said it was essential that no other metal be in contact with the aluminium being treated: such articles had to be disassembled, then reassembled after oxidation, injury to the film being then prone to occur. There was also the difficulty of suspending the article. The appearance of the finish on certain alloys, such as duralumin after heat-treatment, was unsatisfactory, showing up the crystal structure. Did this affect the corrosion resistance? Very good deposits upon aluminium had been applied from time to time; the difficulty was to find a reliable process which would give, say, 90 per cent. satisfactory results.

#### **Effect of Excessive Voltage on the Anode Film**

Mr. J. W. Perring said that the variation in voltage could be carried out smoothly by means of one of the variable speed gears on the market. With regard to the breakdown of the film due to excessive voltage, the same occurred in the Nodon valve, consisting of aluminium in trisodium phosphate solution, where, if the voltage were high, sparks could be seen, the site of which showed pits in the aluminium. With this valve, using tantalum in magnesium sulphate solution, the anodic film could be seen to flake away.

Mr. S. E. Weill said the anodic process could now be satisfactorily worked on both aluminium and duralumin. Coloured finishes could be obtained afterwards without difficulty, and there was no difficulty with the generator. The solution was so cheap that it was best to scrap it periodically. Work should be suspended by aluminium wires secured tightly. The big difficulty in plating upon aluminium was that the latter oxidised so readily.

Mr. S. Wernick (hon. secretary) said that there was no doubt about the efficiency of the anodic process as a method for protecting aluminium. It did not, however, seem necessary to carry out the step-up in voltage in the exact manner suggested, since he had obtained good results using fewer stages of voltage adjustment, and less time in the bath. With regard to nickel plating upon aluminium, he had examined a number of advocated methods some five years ago, but no method gave absolutely satisfactory results. After outlining his experience with these methods, Mr. Wernick said the most promising method made use of an initial coat of iron obtained by a dip in an iron salt.

Mr. Sutton then replied to the discussion, and was accorded a hearty vote of thanks for his interesting paper.

#### **Exploration Work in Canada**

CANADIAN exploration and mining companies are likely to spend as much as two million dollars this year on prospecting activities and elaborate organisations have been set up for this work. About 500 men are being employed by the larger companies, whose work ranges from North Patricia (Ontario), as far north as the Arctic Circle. This number is, of course, additional to the hundreds of prospectors who are sent out by syndicates and those who go out on their own account.

About seventy aeroplanes are now in use by prospecting organisations, and the operations of these are directed by wireless stations maintained at the representative head offices. The Northern Aerial Minerals Exploration Co. has a payroll of 119 men, of which 82 are in the prospecting organisation, these including engineers and prospectors. The company has twelve pilots and student pilots and four mechanics. Dominion Explorers, Ltd., another aerial prospecting organisation, has about 100 men scattered throughout the north country, mainly in north Saskatchewan and in the territory west of Hudson Bay. Other companies engaged in this work have probably from 200 to 250 men on their pay-rolls.

## Metallurgical Topics: Monthly Notes and Comments

From Our Own Correspondents

### The International Congress

THE Sixth International Congress of Mines, Metallurgy and Applied Geology will be held in Liège next year, during the course of the International Exhibition. The opening date will be June 22, 1930, and the Congress will continue until June 30. There will be sections devoted to blast furnaces, steels and ferrous alloys, non-ferrous metals, non-ferrous alloys, etc. Details may be obtained from the Secrétaire Général, Congrès Internationale des Mines, de la Métallurgie et de la Géologie Appliquée, 16, Quai des Etats-Unis, Liège, Belgium.

### The Electroplaters and Depositors

THE annual general meeting which inaugurated the fifth session of the Electroplaters' and Depositors' Technical Society, held last month, bore abundant testimony to the remarkable progress which the Society has made since its foundation five years ago. For some time past it has been publishing its *Transactions* in permanent form, and Volume IV, the latest issue, is a very attractive and informative publication. The Society now has a membership of 300, which shows that it has not nearly exhausted its membership resources, for electrotyping is now, thanks very greatly to the Society's own efforts, a progressive and growing industry, and there must be many hundreds of platers who are eligible for election. The Society has done such useful work, and is engaged on problems of standardisation and on other branches of investigation which will be so beneficial to the industry, that it deserves and should have all the support it needs. At present its subscription income is plainly inadequate, but there must be many who are in the position to join its ranks and so to increase its resources. A donation to its funds would be a good investment for anyone interested in the electrodeposition of metals, or in the allied subject of protective metallic coatings generally. At the meeting above referred to, Dr. Hutton, whose work for the British Non-Ferrous Metals Research Association is so well-known and esteemed, was elected president, and delivered his inaugural address. The coming session will be a busy one, and we wish Dr. Hutton and the Society every success and a substantial increase both in membership and funds.

### Zinc and Gold: Figures of Production

THE recent closing down of yet another spelter works in South Wales serves to illustrate the continued decline in the production of base metals in this country. Tin is the only one now remaining in which Great Britain contributes anything more than an insignificant proportion of the world's total, and even as regards tin, most of the ore is, of course, imported. Yet Belgium, which produces very little zinc ore, produces nearly 16 per cent. of the total zinc output of the world from imported ore, and what can be done there could surely be done here. Precisely why, with Government subsidies to aid the smelting of Australian concentrates, our own spelter industry should so languish is not easy to understand. However, when the projects for the manufacture of electrolytic zinc in the Principality materialise, the situation may improve. The present prices of zinc and lead are not very attractive to British makers, but it may be assumed that they have now reached bed-rock. There has always been about tin a glamour which is perhaps shared by no other metal but gold. The gold production of Great Britain rose during the war to an average of some 1,300 ounces per annum, but has since fallen so greatly as not to be worth recording. On the other hand, the British Empire produces 71 per cent. of all the gold in the world. Those interested in output statistics will find an abundance of information as to gold production in a recent publication of U.S. Department of Commerce, Bureau of Mines, Washington. It is numbered "Economic Paper 6," and is entitled "Summarised Data of Gold Production." In it, amongst other entertaining facts, it will be found recorded that more than half the entire gold production of the whole world, since the discovery of America in the year 1493, has been mined and marketed since the commencement of the present century. Another monograph in the same series deals with zinc ("Economic Paper 2").

### Blast Furnace Practice

WITH the publication of Vol. III, "Operation and Utilisation of Subsidiary Products," which appeared last week, Mr. Fred Clements's great work on *Blast Furnace Practice* (Ernest Benn, Ltd.) has now been completed. The first two volumes, brief notices of which have already appeared in these columns, have had a unanimous welcome in metallurgical journals at home and abroad, and the third will be hailed as in every way worthy of the others. The first volume dealt with the general principles of blast furnace practice, and with the source, preparation, and handling of the raw materials. The second dealt with the design of plant and equipment, and the present volume supplies the working details. These are treated in the methodical manner and in the logical sequence which Mr. Clements's readers have learned to expect. The first chapter explains the principles upon which the calculation of the furnace burden is based, and the methods of calculation employed. These are illustrated by concrete examples of various burdens for the various grades and compositions of pig iron required, and take into consideration an equally wide range of ores, illustrative both of British and of foreign practice. The control of furnace temperature, and the blowing in, damping down and blowing out of a furnace constitute the subject of the next two chapters, which are followed by another dealing with the operations—as apart from design and equipment, dealt with in Vol. II—of accessories such as blowing engines, boilers, hot-blast stoves, etc.

Safety and hygiene, and the economics of labour, are the subjects of the succeeding chapters, after which the keeping of operation records, costing systems, and the description of labour-saving appliances are dealt with. The remaining chapters are devoted to the cleaning and application of blast furnace gas, for heating and power purposes, and the utilisation of flue dust and of blast furnace slag. Finally, two chapters relate to the manufacture of ferromanganese and manganese alloys, and to that of charcoal, pig and electric pig-iron, respectively.

### A Notable Book

SUCH a mere list of chapters fails to give more than a rough idea of the range and treatment of the work, but helps to convey some idea of the thoroughness with which, in the concluding volume of his work, Mr. Clements has handled his subject. It is no exaggeration to say that it is the most important and most exhaustive book that has ever been published in relation to the branch of metallurgy with which it deals, and British metallurgists have every reason to be proud that such a work should have been published in their own country, and by one of their own colleagues. Not only has it the merit of dealing accurately and comprehensively with every aspect of blast furnace practice, but it is eminently readable as well, and succeeds in being not only informative but interesting. Like the previous volume, Volume III is beautifully produced and illustrated, and reflects the greatest credit both on the author and on the enterprising firm by whom it has been published. In all matters the author is a safe guide, and his book is one to which all concerned in blast furnace operation may safely turn for a solution of any of the many problems likely to beset them.

### Rolling Mill Practice

THERE have been no very radical changes in rolling mill practice for a good many years past, such changes as have taken place being related to the improvement of rolling mill accessories and handling appliances, rather than to the actual rolling operation itself. Broadly, as was indicated at a recent open discussion on rolling which took place before the co-ordinating committee meeting in Birmingham last month, the two schools of thought represented respectively by the advocates of slow rolling, with heavy reductions at each pass, and high speed rolling with much lighter reductions per pass, are seeking to find a *via media*, and the tendency is towards quick rolling and heavy passes. The discussion bore more on strip mill practice and methods suited to non-ferrous metals than on heavy plate and section mill practice in steel works, but the methods of rolling metals, are, in both cases, governed

less by what the mill engineer can achieve, than by what the metals will stand—without annealing. The problem differs, moreover, in the case of hot rolling, from what occurs in cold rolling. One of the speakers stated that, within his own experience, 90 per cent. reduction could be effected without annealing, but neither the nature of the metal nor the size of the material was indicated. For the rolling of non-ferrous metals, and for the cold rolling both of non-ferrous and of steel strip, the six-high, or cluster, mill is growing in favour. Up to now, however, although, in point of finish, the work they turn out is excellent, and the power requirements not high, they are not as speedy as other types of mill, and speed, nowadays, is an essential factor of economic production.

## The Demand for More Sections

In heavy steel rolling mills, British practice is being challenged by customers, and reference to this was made in these columns last month in connection with the discussion which took place on a paper read before the Institution of Structural Engineers. As a matter of fact, the subject was revived at an adjourned discussion held subsequently, when a panel was formed to make recommendations on this and allied subjects to the steel committee of the Institution. So far as British steel sections are concerned, complaint is made that building construction is hampered by an insufficient range of sizes. The rolling mills would probably claim, on the other hand, that the selection and choice of building construction sizes in beams, girders, etc., was already wide enough. In America, where a huge home market enables rolling mills to work continuously on a given section, and thus avoid frequent changes of rolls and the ensuing delays, conditions are economically much more favourable already than in great Britain, while the large number of mills at the numerous works in the States enables *all* to be profitably engaged on different sizes, if necessary, and yet work "to capacity."

At home it is quite otherwise; orders are on a much smaller scale, and not only are roll changes frequently involved, but the works already complain of the large number of rolls of varying section that have to be kept in stock. Some of these are required so seldom, or for such small orders, that they involve the locking up of a great deal of capital that can ill be afforded. Rationalisation may, if anything, tend still further to reduce the number of sections available; on the other hand, it might, by careful allocation of rolling quotas amongst the most suitable mills, satisfy the demand of structural engineers for a bigger variety of sections. The problem is a serious one, for if structural engineers establish a "case," and British mills cannot satisfy their requirements, there may be a tendency to place orders for special sections abroad, where they can be got.

## German Cyanide Casehardening Regulations

THE use of cyanides in casehardening has been made the subject of special regulations in Germany, the regulations being issued by the Minister of Health. It is required that cyanide shall be stored in vessels which can be tightly closed, and all storage and other tanks must be plainly marked to show the poisonous nature of the contents. The shop must have level, smooth floors, which must be thoroughly cleaned once each day. The article to be casehardened must be thoroughly dry when placed in the oven. The exhaust hood must fit closely and must not be opened except when the articles to be casehardened are placed in or withdrawn from the boxes or when the bath is to be recharged or emptied. The quenching vessels must be plainly marked "poison," and the quenching water must not under any circumstances be treated with acid. This water must be treated with copperas until a greyish green or blue precipitate is formed, in order to free it from the dangerous soluble cyanide constituents. The worker must wear an apron and gloves of strong materials, and also a mask to protect him from splashes.

## Contract for Roumanian Coinage

THE Royal Mint has recently concluded a contract with the Roumanian Government for the coinage of 50,000,000 twenty-lei and 60,000,000 five-lei pieces. The pieces are to be struck in a new alloy which has recently been experimented with at the Mint and has proved satisfactory. The obverse of both these pieces will show the head of the young King of Roumania. The reverse of the twenty-lei piece shows an allegorical group

representing the old Roumania embracing the newly acquired provinces of Greater Roumania. That of the five-lei piece shows the arms of the new Roumania. A proportion of the pieces will be struck at Birmingham under Royal Mint supervision. The coinage is to be delivered in consignments, the first of which will be made about Christmas-time and the whole completed within six months.

## Preservation of Metal in Sea-Water

THE committee of the Institution of Civil Engineers which is investigating the deterioration of structures in sea-water has issued its ninth interim report, published by H.M. Stationery Office for the Department of Scientific and Industrial Research (3s. 6d. net). The report follows the same lines as its predecessors, being occupied mainly by records of the loss of weight and extent of pitting of specimens of iron and steel exposed in various situations. Dr. J. N. Friend describes the bars which have been exposed for five years at Plymouth and Colombo respectively, the corresponding sets exposed at Auckland and Halifax having been reported on last year. In general, the four sets are in fairly good agreement. Completely immersed specimens show less difference than those exposed to alternate wet and dry conditions, whilst aerial exposure gives the greatest differences between the best and the worst specimens. Nickel has a marked effect in lessening the corrosion of steel, and highly alloyed steels are very resistant, although steel with 13 per cent. of chromium is badly pitted when continuously immersed. The effect of various paints and coatings is still under investigation.

Professor G. Barger reports on the protection of timber by impregnation with arsenical poisons, and finds that low concentrations give insufficient protection against *Teredo*, whilst uniform impregnation is not easily effected. *Limnoria* is proof even against the highly poisonous arsenical preservatives, but certain woods appear to be immune from its attack.

## Electrolytic Zinc Co. Developments

It was recently announced that the Electrolytic Zinc Co., of Australasia, Ltd., has decided to extend its plant at Risdon, Tasmania, to provide additional capacity representing approximately 30 per cent. of that of the existing equipment. The company's output of zinc, which for the year ended June 30, 1929, was 50,000 tons, will thus be increased to about 65,000 tons. This increase in production will become effective in about 18 months' time.

In reaching this decision the directors have been influenced by the desirability of balancing the position that will arise in about two years' time, when the company will have to purchase concentrates in the open market, in addition to those derived from its own mines, at a price in excess of that now being paid under its purchase contracts. The programme of expenditure as at present arranged will not necessitate the raising of additional capital for the extensions.

## Increased Cobalt Output in Canada

AN increase of 76,000 lb. was reported in the Canadian production of cobalt during 1928, the output calculated by the Dominion Bureau of Statistics being 956,590 lb., as compared with 880,590 lb. in 1927. The aggregate value of the output was, however, lower in 1928, being \$1,672,320, as compared with \$1,764,534 in 1927. The Canadian cobalt production was chiefly from the Cobalt district of Ontario, where this metal occurs in association with silver. In addition to the exports from Ontario, the Bureau states that there was a small shipment of ore containing gold, arsenic and cobalt from the province of British Columbia to Australia. The remainder of the world's supply is produced in the Belgian Congo, where cobalt occurs in association with copper.

Canada's exports of cobalt in 1928 were valued at \$1,734,461, or somewhat higher than in the previous year, when \$1,678,468 worth was shipped to other countries in cobalt alloys, metallic cobalt, cobalt oxides, cobalt salts and cobalt ore. Cobalt is used in the form of oxide as a pigment in the manufacture of earthenware, porcelain and enamelware, producing the well-known "cobalt blue" colour. It is also used in the manufacture of driers for paint and varnish. Cobalt metal is chiefly used in manufacture of Stellite, a cobalt-chromium-tungsten alloy for high-speed cutting tools.

## Some Inventions of the Month

### By Our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature, published weekly in THE CHEMICAL AGE.

#### Alloys

THE production of iron alloys containing copper is described by W. P. Digby, of London, in Specification 319,899, dated August 30, 1928. The products contain 10 to 25 per cent. of copper, 0 to 9.5 per cent. of chromium, together with carbon and/or manganese not exceeding 0.8 per cent., the balance being iron. The carbon may be derived from the electrodes of an electric furnace used. The manganese is added about five minutes before the actual pouring takes place.

An electrolytic process for the production of alloys of alkali or alkaline earth metals with lead or other metals is described in Patent Application 318,599, bearing the International Convention date September 6, 1928, by Metallgesellschaft Akt.-Ges., of Frankfurt-on-Main, Germany. Fused halide salts of the alkali or alkaline earth metals are electrolysed with liberation of the metals in the presence of a molten cathode of lead or other metal, the temperature being maintained above 630° C., e.g., at 650° to 700° C., to prevent the formation of disturbing intermediate layers, e.g., of the alloy  $Pb_2Ca$ , at the surface of the cathode. The electrolyte is dehydrated as far as possible before electrolysis.

ALLOYS specially suitable for making wire electrodes of thermionic valves are made by heating a briquetted mixture of a comparatively refractory metal, such as nickel, cobalt, iron, or titanium, in powder form, and a compound of a volatile metal such as an alkali or alkaline earth metal. The production of a nickel-barium alloy from powdered nickel and barium nitride is specified. See Patent Application 317,785, bearing the International Convention date August 22, 1928, by Westinghouse Electric and Manufacturing Co., of East Pittsburgh, U.S.A.

#### Electrolytic Extraction of Metals

METALLIC elements, other than the alkali metals, are obtained by electro-deposition from solutions of salts of the metals in liquid ammonia. Suitable salts are chlorides, bromides, iodides, nitrates, cyanides and sulphocyanides. The process is specially advantageous in that it permits of electrolytic production of metals, which are not obtainable by electrolysis of their aqueous solutions. Such metals are beryllium, tungsten, tantalum and molybdenum, the salts of which hydrolyse in contact with water, with formation of insoluble or sparingly soluble basic salts and magnesium, the ions of which react at the cathode with the solution and form basic compounds. The process is applicable to the purification of the metals by using anodes of the impure metals, and an electrolyte comprising a solution of a suitable salt, e.g., an ammonium salt, in liquid ammonia. See Specification 320,818, dated December 31, 1928, by L. St. C. Broughall, of Barnet, Herts.

#### Extracting Metals

THE oxide constituent of a thermo-aluminic mixture is in the form of porous grains, e.g., of crushed sinter. Thus iron ores, fine borings, mill scale, or other iron oxide materials are crushed, screened, mixed with a small proportion of fuel, moistened, sintered in a thin layer by blast-roasting, and mixed with finely divided aluminium. If desired, additional iron may be melted down by the reaction. Oxides of chromium and vanadium are similarly reduced by means of aluminium, calcium, or other reducing metals. See Patent Application 319,775, bearing the International Convention date September 29, 1928, by R. W. Hyde, of Summit, New Jersey, U.S.A.

#### Manganese

IN Specification 319,605 (Dicker), a communication from Bradley-Fitch Co., of Minneapolis, Minnesota, U.S.A., dated June 19, 1928, a process is described for recovering manganese values from materials containing manganous oxide, e.g., materials obtained from manganese-iron ores by the treatment described in Specification 284,098 (See THE CHEMICAL AGE, vol. xviii, p. 182). The leaching of the materials is effected with a solution of one or more ammonium salts, such as the sulphate, in considerable excess of the quantity corresponding theoretically to the manganese present. Preferably the solution is used at a temperature approaching the boiling point, and at a pressure below atmospheric. The leaching is

carried out in shallow vessels, such that at ordinary pressure the liquid has a depth of about 18 inches, the depth increasing in proportion to the decrease of external pressure, so that the solubility of the ammonia is not increased in the lower strata of the liquid beyond the value at which interference with the process and precipitation of the manganese occurs. The subject matter of the specification has been divided out from Specification 319,392. (See THE CHEMICAL AGE, vol. xxi, p. 37 [Metallurgical Section]).

#### Precious Metals

A PROCESS for the recovery of gold, silver and metals of the platinum group by amalgamation methods is described in Specification 320,185, dated August 20, 1928, by D. Enzlin, of Johannesburg, and J. A. Eklund of Pietersburg, Transvaal, South Africa. The metalliferous material, mixed with an activating solution (comprising hydrochloric acid, one or more chlorine compounds of mercuric mercury, and zinc chloride,) is brought into contact with zinc amalgam or tin amalgam. The presence of free chlorine and/or sodium chloride in the mixture is advantageous in some cases. The specification contains a discussion of the probable nature of the various reactions occurring in the process, and examples are given of the treatment of (1) a pyritic gold ore; (2) a gold ore containing grey copper, antimony, and arsenic; (3) a gold ore containing antimony and arsenic, but little copper; (4) a sulphide platinum ore; and (5) an oxidised platinum ore. The invention permits the recovery of the metals directly from their ores without preliminary concentration.

#### Production of Metals

THE carbon monoxide liberated in the production of metals (e.g., iron) by the thermal decomposition of their carbonyl compounds is unsuitable for use in the production of further quantities of carbonyl compounds in that it produces erratic rises of temperature which damage the apparatus and impair the reactivity of the metals. This difficulty is obviated in an invention described in Specification 320,177 (Johnson), dated August 13, 1928, a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany, by freeing the carbon monoxide from admixtures which promote its decomposition into carbon and carbon dioxide or which hinder the production of the carbonyls by diminishing the reactivity of the metals. Thus, the carbon dioxide present and the fine metallic dust suspended in the gas may be removed by means of scrubbing liquids or solid absorbents, or the metallic dust may be precipitated electrically.

#### Refining Metals

SCRAP metal, e.g., copper or zinc, is refined by forming it into briquettes, subjecting these at a temperature below the melting point of the metal to the action of a substance such as hydrogen or water gas which removes the impurities from and cleans the surfaces of the metals, and finally extruding the cleaned metal through a hollow plunger. Nitrogen, steam, carbon dioxide, or hydrocarbon gases may be present with the hydrogen or water gas. See Patent Application 317,400, bearing the International Convention date August 15, 1928, by H. H. Stout, of Douglas, Arizona, U.S.A.

#### Treatment of Ores

A PROCESS for the treatment of ores or the like containing acid-oxide forming metals, such as chromium, manganese, molybdenum, titanium or uranium, is described in Specification 320,845, dated April 21, 1928, by R. W. Stimson, of New York, U.S.A., the object being to produce valuable salts such as bichromates, chromates, permanganates, or titanates, together with useful by-products. An intimate mixture of the powdered or granulated ore with a sufficiency of basic material to form a salt with the acid oxide of the metal is heated to a temperature not exceeding about 700° C., while maintaining the requisite oxidising conditions by means of nitrogen peroxide supplied from an external source. The nitric oxide emerging from the reaction zone may be converted into the peroxide for use again. The salts may be extracted from the reaction mixture by leaching with dilute sulphuric acid produced by means of some of the nitric oxide evolved. In the case of the treatment of a chrome iron ore the solid residue from the leaching is a porous granular mass of substantially pure  $Fe_2O_3$ , admixed with the gangue of the original ore, and is suitable for briquetting for use in blast furnaces, for the formation of cast iron and cement-forming slag.

## Current Articles Worth Noting

We give below a brief index to current articles in the technical Press dealing with metallurgical subjects.

**ALLOYS.**—The production of the  $\beta$ -phase of silver-zinc alloys. B. G. Petrenko. *Zeitschrift analytische Chem.*, Vol. 184, Part 4, pp. 369–375 (in German).

The electrical conductivity of silver-zinc alloys in the annealed condition. G. J. Petrenko. *Zeitschrift analytische Chem.*, Vol. 184, Part 4, pp. 376–384 (in German).

The system copper-zinc. R. Ruer and K. Kremers. *Zeitschrift anorganische Chem.*, Vol. 184, Parts 1–3, pp. 193–231 (in German). From the fusion diagram and the properties of the individual alloys, it is deduced that two copper-zinc compounds exist, of which one lies in the  $\gamma$  region and contains about 40 per cent. of copper, while the other lies in the  $\beta$  region and contains about 51 per cent. The formula of the first compound is, if not  $\text{Cu}_2\text{Zn}_3$  (which requires 39.32 per cent. of copper),  $\text{Cu}_{21}\text{Zn}_{31}$  (which requires 39.7 per cent., and, in contrast with the simple formula, is consistent with the lattice structure of  $\gamma$ -brass). In consideration of the lattice structure of  $\beta$ -brass, the simplest formula for the second copper-zinc compound is  $\text{Cu}_{14}\text{Zn}_{13}$ , which requires 51.14 per cent. of copper.

Effect of small quantities of third elements on the ageing of lead-antimony alloys. E. E. Schumacher, G. M. Bouton and L. Ferguson. *Ind. Eng. Chem.*, November, pp. 1042–1044.

The effect of third metals on the constitution of brass alloys. II.—The effect of nickel. O. Bauer and M. Hansen. *Zeitschrift Metallkunde*, November, pp. 357–367 (in German). Deals with the ternary system nickel-zinc-copper; and with the hardness of nickel brasses.

**ANALYSIS.**—The sampling and analysis of red brass and other alloys inclined to liquation. O. Bauer and E. Deisz. *Zeitschrift analytische Chem.*, Vol. 79, Part 1–2, pp. 47–53 (in German).

The volumetric determination of carbon in iron and steel alloys difficult of combustion. E. Zindell. *Chemiker-Zeitung*, November 16, p. 891 (in German).

**COPPER.**—The system copper-oxygen. R. Vogel and W. Pocher. *Zeitschrift Metallkunde*, October, pp. 333–337; November, pp. 368–371 (in German). Deals with the phase diagram of the system copper-copper oxide, especially with the solubility of oxygen in copper and the miscibility gap in the copper-copper oxide area. The decomposition of cuprous oxide at low temperatures is also discussed.

**CORROSION.**—Investigations on the corrosion of aluminium and duralumin in sea-water. Report of the German Metallurgical Society. *Zeitschrift Metallkunde*, November, pp. 378–386 (in German).

The corrosion of brass. II.—H. Masukowitz. *Korrosion und Metallschutz*, October, pp. 217–226 (in German).

Corrosion and the protection of metals in boiler operation. II.—R. Stumper. *Korrosion und Metallschutz*, October, pp. 230–235 (in German).

**ELECTRODEPOSITION.**—The effect of addition agents upon conductivity, cathodic polarisation and grain size of deposits obtained from the cell:  $\text{Cu}/\text{CuSO}_4, \text{H}_2\text{SO}_4/\text{Cu}$ . B. Clark and E. O. Jones. *Trans. Faraday Soc.*, November, pp. 583–590. The use of addition agents (gelatin, dimethylaniline, etc.) results in all the cases investigated in an increase of cathodic polarisation, decrease in the conductivity of the electrolyte, and reduction in the grain size of the deposit.

The electrodeposition of silver from argentocyanide solutions. S. Glasstone and E. B. Sanigar. *Trans. Faraday Soc.*, November, pp. 591–598. Discusses the effect of the presence of various anions on various aspects of the deposition.

The electrolytic coating of metals with chromium. W. Birett. *Zeitschrift Metallkunde*, November, pp. 372–377 (in German).

**INHIBITORS.**—Inhibitors in the action of acid on steel. F. H. Rhodes and W. E. Kuhn. *Ind. Eng. Chem.*, November, pp. 1066–1070. Measurements were made to determine the efficiencies of several organic compounds of nitrogen

as inhibitors in the action of sulphuric acid on iron. The most efficient inhibitors were cyclic compounds which contained an atom of nitrogen in the ring. In cyclic compounds of this type the inhibiting effect increases with the molecular weight. The introduction of methyl, phenyl, or amino groups or the addition of hydrogen increases the inhibiting action. The most efficient inhibitors found were derivatives of acridine containing several substituted methyl and amino groups. The addition of inhibitors of this type to dilute sulphuric acid in contact with iron increases the interfacial resistance between the acid and the iron. There is no quantitative relationship between the increase in interfacial resistance and the inhibiting effect, so that it appears that adsorption on the surface of the metal is a necessary condition for inhibiting action, but that the extent of the inhibiting effect depends also upon some specific property of the adsorbed film. Experiments were made which show that the inhibitor is actually adsorbed on the surface of the metal.

**IRON.**—Equilibrium investigations in the reduction, oxidation and carburisation of iron. IX.—The effect of other oxides on the equilibrium position. R. Schenck, H. Franz and H. Willeke. *Zeitschrift anorganische Chem.*, Vol. 184, Parts 1–3, pp. 1–38 (in German).

Observations on the iron-nitrogen system. S. Epstein, H. C. Cross, E. C. Groesbeck and I. J. Wymore. *Metals and Alloys*, October, p. 152.

The behaviour of iron towards nitric acid. Z. C. Mutaftschiew. *Zeitschrift Elektrochem.*, November, pp. 861–862 (in German).

**ZINC.**—The electrolytic production of zinc. G. Eger. *Chemiker-Zeitung*, November 6, pp. 857–858; November 13, pp. 878–879 (in German).

## Commercial Intelligence

The following are taken from printed reports, but we cannot be responsible for any errors that may occur.

## Mortgages and Charges

[NOTE.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debts due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case, the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.]

**BOLTON (THOMAS) AND SONS, LTD.**, Widnes, copper manufacturers. Registered November 12, £50,000 debentures, part of amount already registered; general charge. £500,000. August 1, 1928.

**CYPRUS SULPHUR AND COPPER CO., LTD.**, London, S.W. Registered November 7, £2,600 1st debentures, part of £20,000; general charge. \*£10,651. June 26, 1929.

**MILLS (S.) AND CO., LTD.**, Birmingham, brassfounders. Registered October 30, £1,000 charge, to Eliza A. Bryden, 32, Oxtou Road, Birkenhead, retired publican; charged on land and buildings in Sycamore Road, Handsworth, Birmingham. \*£4,000. August 9, 1928.

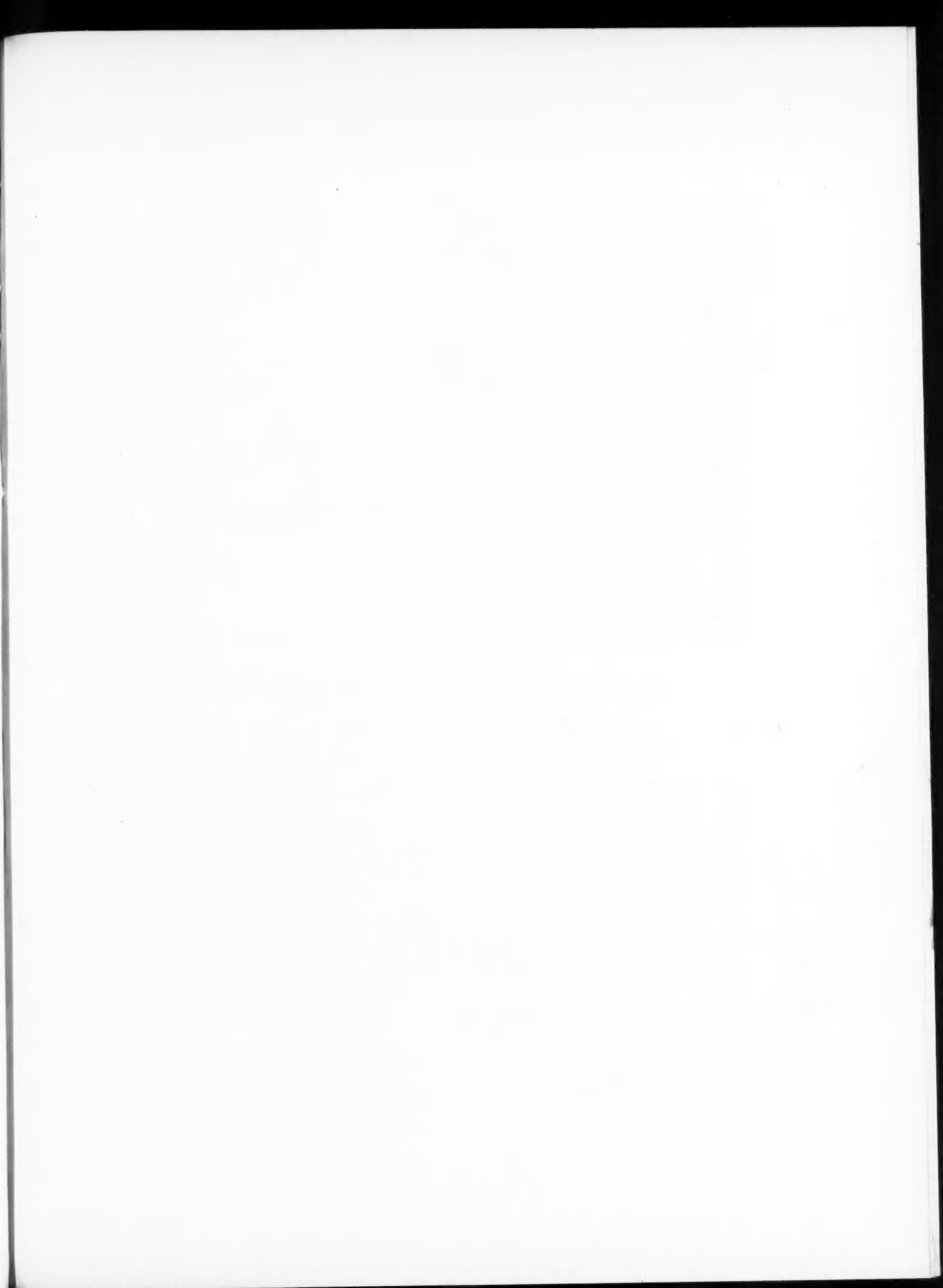
**SHEPHARD (W. H.), LTD.**, Sheffield, steel manufacturers. Registered November 15, £3,000 debentures, to Hallamshire Steel and File Co., Ltd., Neepsend; general charge. \*£4,700. December 13, 1927.

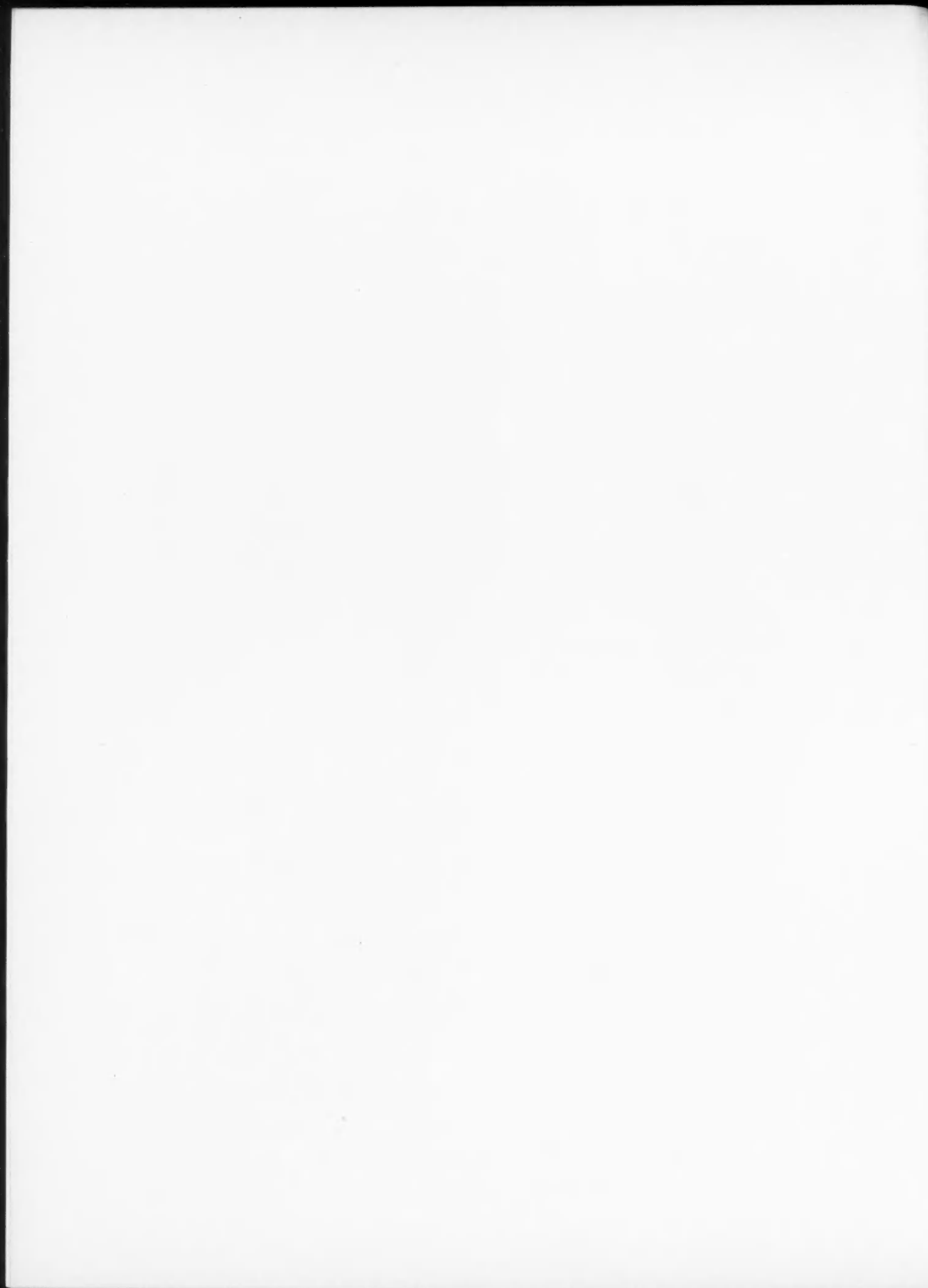
**WIGPOOL COAL AND IRON CO., LTD.**, Gloucester. Registered October 17, £500 debentures, part of amount already registered; general charge. \*£26,000. April 4, 1929.

## Satisfactions

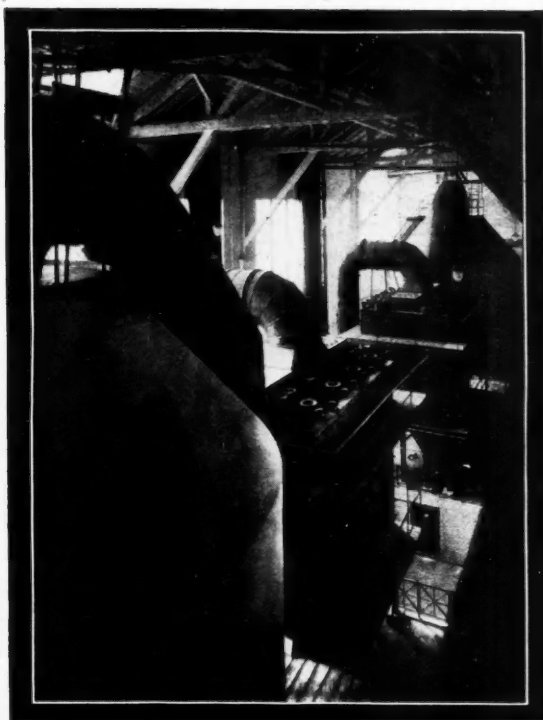
**BWANA M'KUBWA COPPER MINING CO., LTD.**, London, E.C. Satisfaction registered October 22, £31,050, part of £1,000,000, etc., registered April 13, 1928.

**SHEPHARD (W. H.) LTD.**, Sheffield, steel manufacturers. Satisfaction registered November 12, £5,000, registered December 30, 1925.





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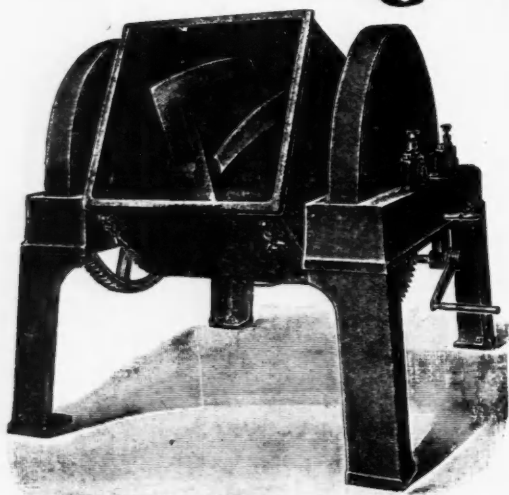
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## INDEX TO ADVERTISERS

- Accurate Recording Instrument Co., Ltd. ....vii  
 Adamson (D. J.) & Co., Ltd. ....xii, Dec. 7  
 Alfred, Shirley & Co., Ltd. ....Ixxiii  
 Alfa-Laval Co., Ltd. ....xxxiii, Nov. 30  
 Alldays & Onions, Ltd. ....xvii, Nov. 30  
 Allen (Athole G.) & Co. ....Ixxvii  
 Allen (Edgar) & Co., Ltd. ....xvii, Dec. 7  
 Alluvial Mining Machinery Co., Ltd. xvii, Dec. 7  
 Aluminium Plant & Vessel Co., Ltd. xiv, Dec. 7  
 Animal Products Co., Ltd. ....Ixxvii  
 Antimony Products Co., Ltd. ....ii, Nov. 2  
 Arnico International Corporation  
     *Front Cover*, Sept. 7  
 Ashdown (H. E.) (B'ham), Ltd. ....xiv, Dec. 21  
 Averill, (C. R.) Ltd. (Sole Agents for Jos.  
     Dixon Crucible Co.) ....x, Dec. 14  
 Bagshawe & Co., Ltd. ....v  
 Baillière, Tindall & Cox ....xxxvi, Nov. 30  
 Baker, Perkins, Ltd. ....Ixxiii  
 Bamag-Meguinn (Great Britain), Ltd.  
     *Cover ii* Nov. 16  
 Banco Commerciale Italiana ....iv, Dec. 21  
 Barclays Bank, Ltd. ....xix, Nov. 16  
 Barter Trading Corporation, Ltd. ....Ixiv  
 Battersea Polytechnic ....iv, Sept. 7  
 Beaumont's, Ltd. ....xvi  
 Belgian State Railway ....xviii, Dec. 21  
 Bell Bros. (Manchester), Ltd. ....lix  
 Benn Brothers, Ltd. ....xii, Nov. 16  
 Benn (Ernest), Ltd. ....xx, xxviii, xxix, Nov. 30  
 Bennett & Jenner, Ltd. ....xli, Sept. 25  
 Best (Jas.) & Sons ....xvii, Dec. 21  
 Birmal Chemical Engineers ....xlvii  
 Blagden (Victor) & Co. ....583, Dec. 21  
 Blyth (H.) & Co. ....xv, Dec. 21  
 Booth (J.) & Sons ....xvi, Sept. 28  
 Boots Pure Drug Co., Ltd. ....iii, Dec. 21  
 Bradford Technical College ....xxxii, Sept. 28  
 Brandram Bros. & Co., Ltd. ....iv, Sept. 7  
 Bright (John) & Bros., Ltd. ....xxv, Dec. 14  
 British Commercial Gas Association ....xxii  
 British Drug Houses ....xxx & Ixvi  
 British Industrial Solvents, Ltd. (The)  
     *Front Cover*, Nov. 9  
 British Industries Fair ....v, Dec. 7  
 British Chemical Plant Manfrs. Assn. ....xxx  
 British Carbo Union Co. ....627  
 British Quadruplex Pumps, Ltd. ....xiv, Aug. 10  
 British Resistor Co. ....*Cover iii*  
 British Steam Specialties, Ltd. ....lv  
 Broadbent (Thos.) & Sons, Ltd. ....Ixvi  
 Brotherton & Co., Ltd. ....Ixiv  
 Brough (E. A.) & Co. ....xv, Dec. 21  
 Brown & Son (Alcembic Works), Ltd. ....Ixxvii  
 Buffoline Noiseless Gear Co., Ltd. xiv, Aug. 3  
 Bueckhardt Engineering Works ....xv  
 Burditt & Co. (1928), Ltd. ....Ixxvii  
 Burt, Boulton & Haywood, Ltd. ....629  
 Butters (Chas.) & Co. ....xxx, Nov. 30  
 Cafferata & Co., Ltd. ....xi, Dec. 21  
 Carter (J. Harrison), Ltd. ....ii  
 Carty & Son, Ltd. ....*Cover iii*  
 Chance Bros., Ltd. ....lii  
 Chesterfield Tube Co., Ltd. ....ii, Dec. 21  
 Christopherson (Clifford) & Co. ....xi, Dec. 21  
 City of Manchester Education Committee. Ixvii  
 City of Salford Education Committee xxxviii  
 Clark (Geo.) & Sons (Hull), Ltd. ....xxviii, Sept. 28  
 Cleckheaton Eng. & Motor Co., Ltd.  
     xxxiv, Sept. 28  
 Clifford (Chas.) & Sons, Ltd. ....xlv  
 Cole & Wilson ....xxxiv, Sept. 28  
 Collett (J. M.) & Co., Ltd. ....Ixxvii  
 Compagnie Commerciale du Nord ....Ixxvi  
 Cook (Chas. W.) & Sons ....xii, Sept. 21  
 Cortin & Corking, Ltd. ....Ixxiii  
 Crane (Fredk.) Chem. Co. ....xlv  
 Crane & Taylor, Ltd. ....xxvi, Dec. 14  
 Curtis, Algernon Lewin  
     Cuthbertson (Jas.) & Co., Ltd. ....xi, Dec. 21  
 Dale (John) Mfg. Co. ....iii  
 Derby & Co., Ltd. ....Ixxii  
 Dextine, Ltd. ....x, Nov. 30  
 Dickinson (Arthur), J., Ltd. ....xxxviii  
 Dickinson (John) Ltd. ....Iviii  
 Dorking GreyStone Lime Co., Ltd. xxviii, Dec. 14  
 Dorr Co., Ltd. ....xxxii  
 Durastic Bituminous Paint Co. ....xvi, Dec. 21  
 Emco Dyes, Ltd. ....xxiv  
 English China Clays, Ltd. ....xviii, Dec. 21  
 Evans, Adlard & Co., Ltd. ....xvi, Dec. 21  
 Evans (R. H.) & Co. ....*Front Cover*, Dec. 21  
 Eversal Products, Ltd. ....xvii, Sept. 14  
 Feltham (W. H.) & Son ....iii  
 Fiechter (Emil) ....xiv  
 Firth (Thos.) & Sons, Ltd. ....xxv  
 Foamite Firefoam, Ltd. ....xxix  
 Follows & Bate, Ltd. ....xxvii  
 Ford (T. B.) Ltd. ....*Front Cover*, Dec. 14  
 Foster Instrument Co. ....xii, Dec. 21  
 Foyle (W. & G.) ....Ixxiii  
 Fraser (W. J.) & Co., Ltd. ....i  
 Gardner (Win.) & Sons ....iii, Dec. 21  
 Gas Light & Coke Co. ....xxxiv  
 General Chemical and Pharmaceutical Co.,  
     Ltd. (The) ....lix  
 Glenboig Union Fire Clay Co., Ltd. xvii, Dec. 21  
 Graesser-Monsanto Chemical Works, Ltd. ....xvii  
 Greeff (R. W.) & Co., Ltd. ....lv  
 Griffiths Bros. & Co. (London), Ltd. ....vii  
 Guthrie & Co. ....*Cover ii*, Dec. 21  
 Haller & Phillips, Ltd. ....Ixx  
 Harris (P. L. & G. S.), Ltd. ....*Cover iii*, Dec. 14  
 Harrison Blair & Co., Ltd. ....viii & Ixvi  
 Haughton's Patent Metallic Packing Co.,  
     Ltd. ....lix  
 Hauwel Filter Cloths. ....xxxiv, Nov. 30  
 Hickson (E.) ....Ix  
 Hill-Jones (Thos.), Ltd. ....Iviii  
 Holland (Bernard) & Co. ....x  
 Holmes (W. C.) & Co., Ltd. ....liii  
 Holst (K.) & Co. ....ii  
 Honeywill (C. & W. R.) ....Ixxvii  
 Hopkin & Williams, Ltd. ....Ixxvii  
 Horne Engineering Co. ....Ixxii  
 Howards & Sons, Ltd. ....515, Nov. 30  
 Huntington, Heberlein & Co. ....xxxii, Nov. 30  
 Hurrell (G. C.) & Co. ....xxxii  
 Hyde (Silas) ....xlv  
 Imperial Chemical Industries, Ltd. ....xi  
 Institute of Chemistry ....Ixxix  
 Integra Co., Ltd., The ....xi  
 International Combustion Co. ....xviii & xix  
 International Correspondence School, xiii, Nov. 23

FOR CLASSIFIED INDEX TO TRADES SEE PAGES lii &amp; liii

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## INDEX TO ADVERTISERS—Continued

Jenkins Bros., Ltd. ....	xx, Dec. 14	Negretti & Zambra .....	Cover ii	Spence, Peter, & Sons, Ltd. ....	xxi, Dec. 14
Jenkins (R.) & Co., Ltd. ....	ii, Dec. 7	Nordac, Ltd. ....	ii, Dec. 7	Spencer, Chapman & Messel, Ltd. ....	xxxvii, lxvi
Jepson (E. G.) & Co. ....	xxxii, Sept. 28	Northern Media Supply. ....	xxvi, Nov. 9	Statham (John) & Sons. ....	Cover ii, Dec. 7
Johnson (C. H.) & Sons, Ltd. ....	xliv, Nov. 30			Staveley Coal & Iron Co., Ltd. ....	ix
Johnson (G. T.) & Co. ....	Cover, ii	Oertling (L.), Ltd. ....	xxxv	Steel Barrel Co., Ltd. ....	lxx
Johnson, Matthey & Co., Ltd. ....	lxxii	Ohlenschlager Bros. ....	lxxii	Steel (J. M.) & Co., Ltd. ....	Cover iii, Dec. 7
Johnson (S. H.) & Co., Ltd. ....	Cover, iv	Oliver United Filters, Inc. ....	xxi	Steward (J. H.), Ltd. ....	lxxix
Johnson (W. F.) & Co. ....	x, Dec. 21	Ougree Steel Trading Co., Ltd. ....	liv	Stewarts & Lloyds, Ltd. ....	xlui
Jones (E. H.) (Machine Tools), Ltd. ....	xxxviii			Stott (James), Ltd. ....	xxii, Dec. 14
Jones, Ivor R. ....	lxvii	Page (Chas.) & Co., Ltd. ....	lxvii	Talbot-Stead Tube Co., Ltd. ....	xlxx
Keay (E. C. J.), Ltd. ....	xlvi	Paterson Engineering Co. ....	lxiv	Tennant (C.) & Co., Ltd. ....	lxxii
Kelvin, Bottomley & Baird, Ltd. ....	lxx	Perry & Hope, Ltd. ....	x, Dec. 21	Tennant (C.) Sons & Co., Ltd. ....	xxvi
Kestner Evap. & Eng. Co., Ltd. ....	viii	Petersen, Anton & Henius, Ltd. ....	xxv, Nov. 30	Thermal Syndicate, Ltd. ....	631
Key Engineering Co. ....	xxii, Dec. 14	Pitman (Sir Isaac) & Sons ....	liv	Thompson Bros. (Bilston), Ltd. ....	xlvi
Kraft Sacks, Ltd. ....	lxx	Pneumatic Conveyance and Extraction (1929), Ltd. ....	ii, Dec. 21	Thompson, John (Dudley), Ltd. ....	xlvi
		Polytechnic, The Northern. ....	xxxvii, Sept. 28	Thornton (A. G.), Ltd. ....	xxii, Dec. 14
Lancaster & Tonge, Ltd. ....	ix	Potter (F. W.) & Soar, Ltd. ....	ix	Tileman & Co., Ltd. ....	Cover ii, Nov. 9
Laporte (B.), Ltd. ....	xxxiv	Power Gas Corporation, Ltd. ....	Cover iv, Dec. 7	Tipton Tub & Tube Co., Ltd. ....	li
Lea Recorder Co., Ltd. ....	xxii, Dec. 14	Pragos Engineering Co. ....	lxv	Todd Bros. ....	xxxii, lxvii
Leeds & Bradford Boiler Co., Ltd. ....	lxix	Premier Filterpress Co., Ltd. ....	xxxvi	Tranter, W. & J., Ltd. ....	li
Le Grand, Sutcliffe & Gell, Ltd. ....	v, Dec. 21	Price, Stutfield & Co., Ltd. ....	Front Cover	Trussed Concrete Steel Co., Ltd. ....	lxxi
Leigh (R. & H.), Ltd. ....	Cover ii, Dec. 14	Prodorite, Ltd. ....	xii, Dec. 21	Tungstone High Pressure Machine Die Casting Co. ....	vii, Nov. 23
Leitch (J. W.) & Co., Ltd. ....	vi	Pulsometer Engineering Works. ....	lxvii	Tyrer (Thos.) & Co., Ltd. ....	xx
Lennox Foundry Co., Ltd. ....	xvi				
Lewis (H. K.) & Co., Ltd. ....	xvi, Nov. 30	Raschig (Dr. F.) ....	ix, Dec. 7	Valor Co., Ltd. ....	1
Lilleshall Co., Ltd. ....	lxi	Reads, Ltd. ....	ix, Dec. 14	Visco Engineering Co., Ltd. ....	xxxix
Limmer & Trinidad Lake Asphalt Co., Ltd. ....	lxviii	Reynolds & Branson, Ltd. ....	xxxii, Sept. 28		
		Richards (E.) & Co. ....	xv, Dec. 21	Walker (James) & Co., Ltd. ....	iv, Dec. 14
McGraw-Hill Publishing Co., Ltd. ....	xxxiii	Rissik Fraser & Co., Ltd. ....	xxx, July 27	Wallach Bros. ....	xxvi
MacKechuie (R. D.) & Co. ....	Cover ii, July 27	Rowlandson (W.) & Co. ....	lx	Walters (Vincent) ....	lxxiii
Maempel (E. O.) ....	xxxii, Nov. 30	Royal Metal Trades Pension & Benevolent Society ....	xx, Dec. 7	Warrington Chem. & Drug Co., Ltd. ....	xi, Dec. 21
Manchester Aluminium Co., Ltd. ....	lv	Rubber Asbestos & Belting Co. ....	xxiv, Dec. 14	Washington Chemical Co., Ltd. ....	xxiii
Manlove, Allott & Co., Ltd. ....	Cover iii, Dec. 21	Ruston & Hornsby. ....	lix	Watts, Hardy & Co. (1920), Ltd. ....	xxviii, Oct. 5
Martineau & Smith ....	iv, Dec. 21			Waxed Bags, Ltd. ....	xxviii
Meldrums, Ltd. ....	xiv & lxvi	St. Helens Cable & Rubber Co., Ltd. ....	xii, Aug. 31	Webb, W. A., Ltd. ....	lv
Metafilters, Ltd. ....	xxi, Nov. 30	Scott (G.) & Son (London), Ltd. ....	xv, Dec. 14	Weinreb, F. ....	lxvii
Metallisation Sales, Ltd. ....	xiii, Dec. 14	Scottish Dyes, Ltd. ....	v	Wengers, Ltd. ....	lxxviii
Metals Coating Co., Ltd. ....	xiii, Dec. 14	Selden Co. (The) ....	xi, Oct. 19	Western Chem. Co. (Paisley), Ltd. ....	lxx
Metcalf & Co. ....	lxxiii	Shigham & Co., Ltd. ....	xxviii, Sept. 28	Westinghouse Brake & Saxby Signal Co. ....	xxviii
Methylating Co., Ltd. ....	vi	Siebe, Gorman & Co., Ltd. ....	lx	West's Gas Improvement Co., Ltd. ....	lxxiii
Metropolitan Vickers Electrical Co. ....	xxxvi	Silica Gel, Ltd. ....	xxi, Dec. 7	Willcox (W. H.) & Co., Ltd. ....	xxii, Dec. 14
Michaud (Wm. J.) & Co. ....	xviii, Nov. 30	Simon (R.) & Sons, Ltd. ....	lxvii, lxix	Wilkinson (James) & Son, Ltd. ....	xxxviii
Mitchell (L. A.) ....	Cover iv	Sloog, H. ....	v, Sept. 28	Wolfe, Keene & Co. ....	lvi
Mond-Nickel Co., Ltd. ....	xx, Sept. 7	Sofnol, Ltd. ....	lxix	Woolley (Jas.) Sons & Co. ....	Cover ii
Monel-Weir, Ltd. ....	xx, Dec. 7	Solvent Products, Ltd. ....	1, Dec. 7		
Mosse (Rudolf) Advertising ....	lxviii			Yorkshire Tar Distillers, Ltd. ....	xxxv, Sept. 28

FOR CLASSIFIED INDEX TO TRADES SEE PAGES lii &amp; liii

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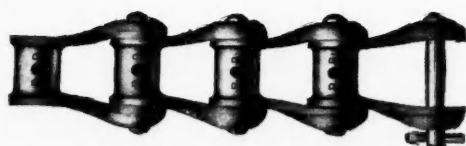
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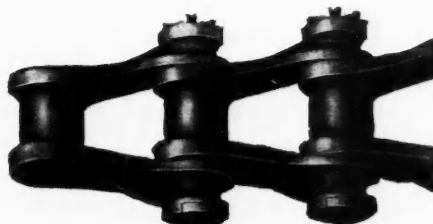
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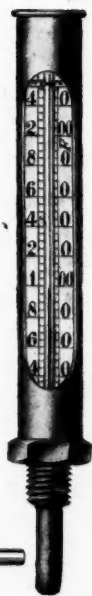
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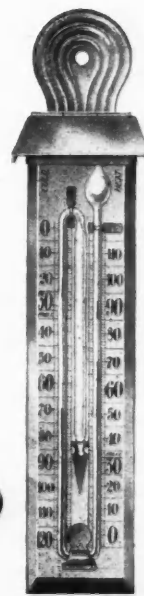
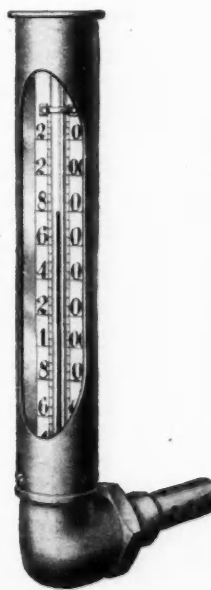


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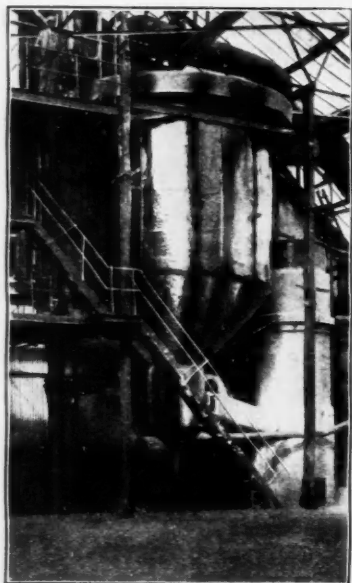
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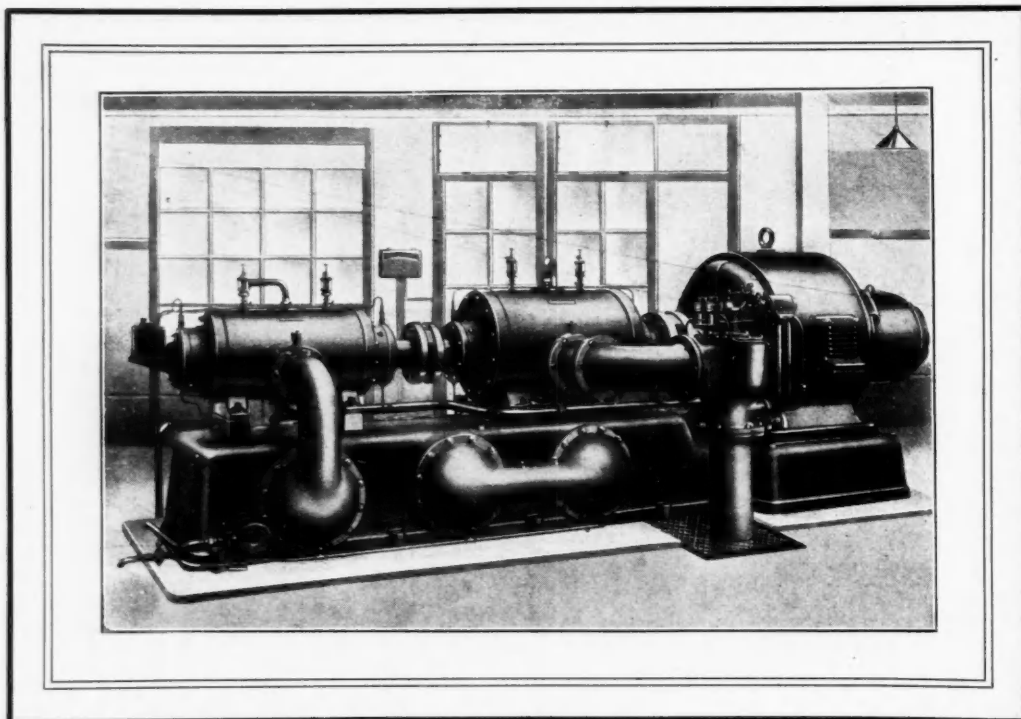
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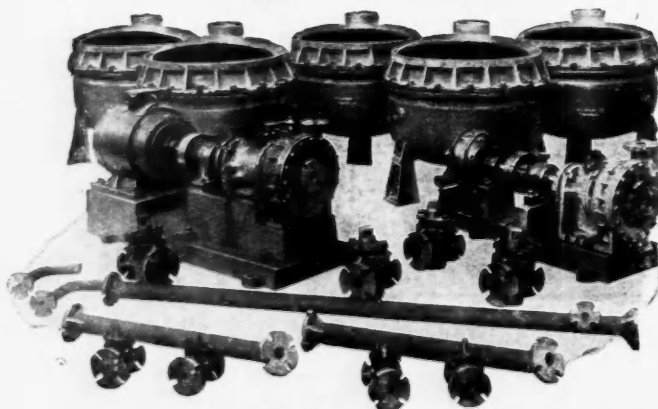
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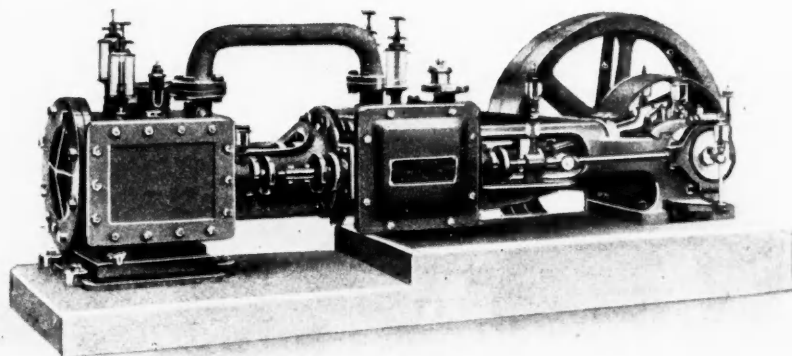
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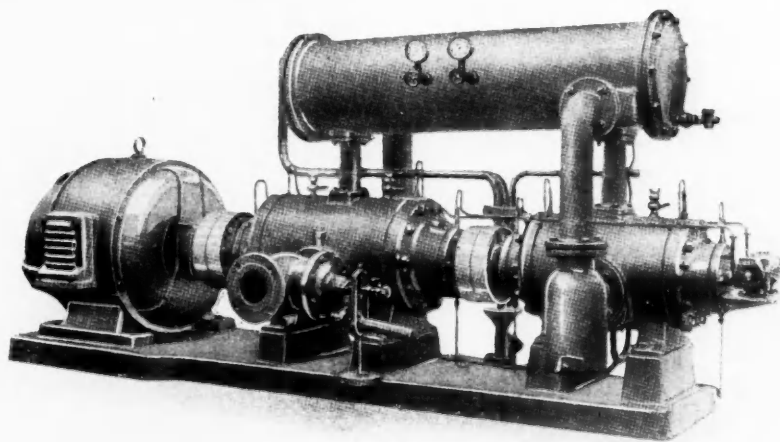
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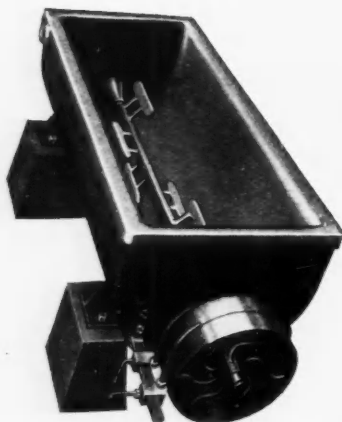
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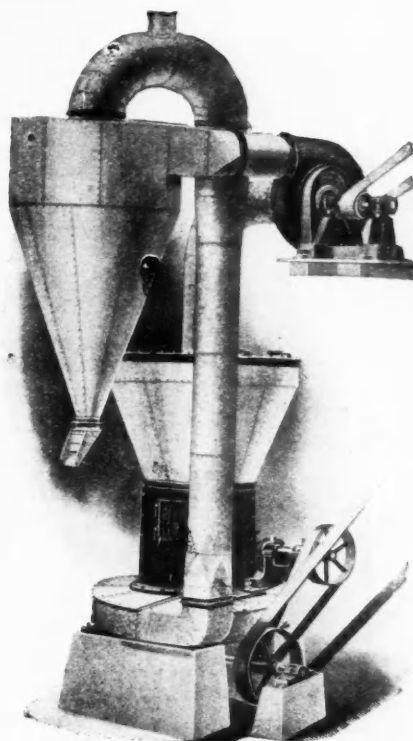
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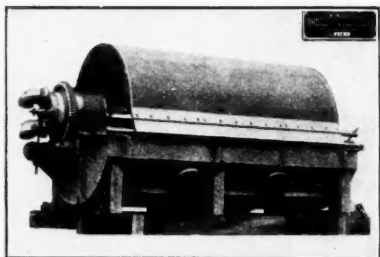
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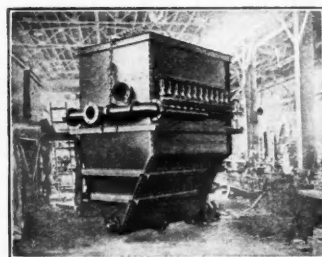
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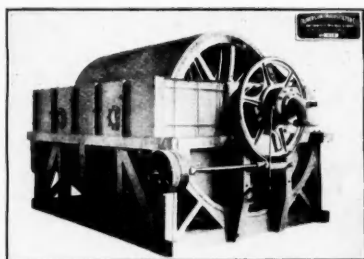
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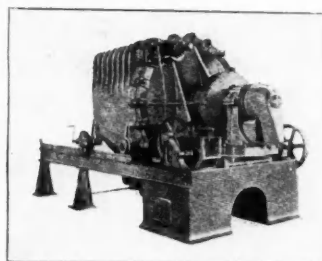
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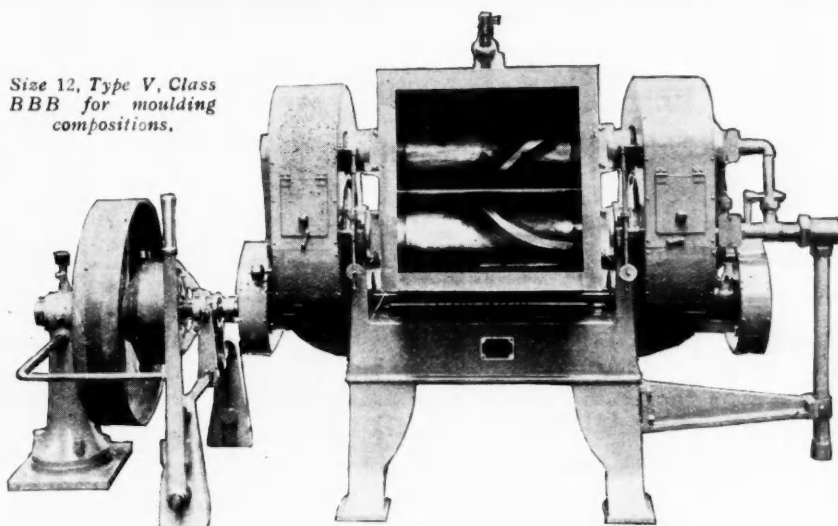
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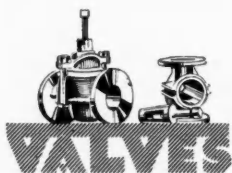
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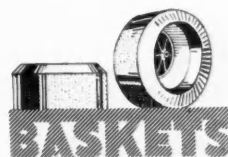
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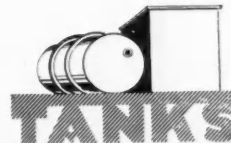
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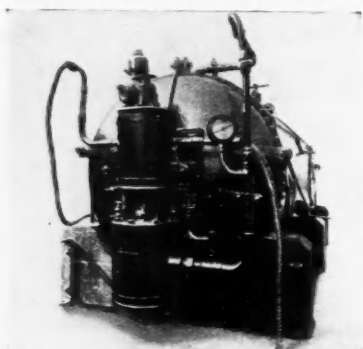
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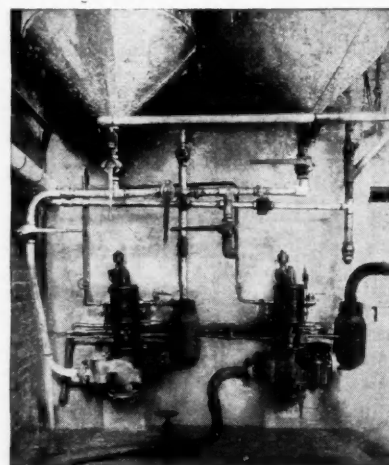


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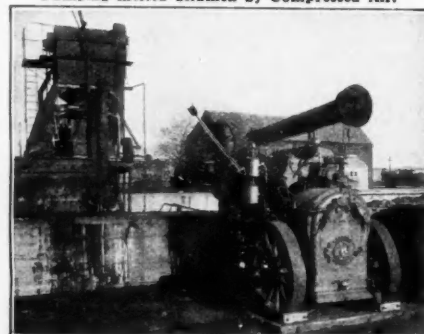
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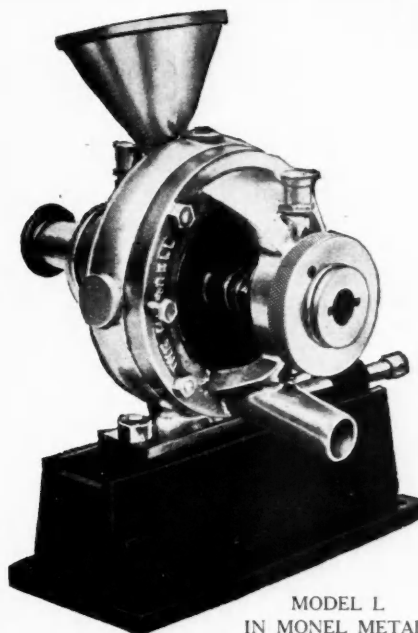
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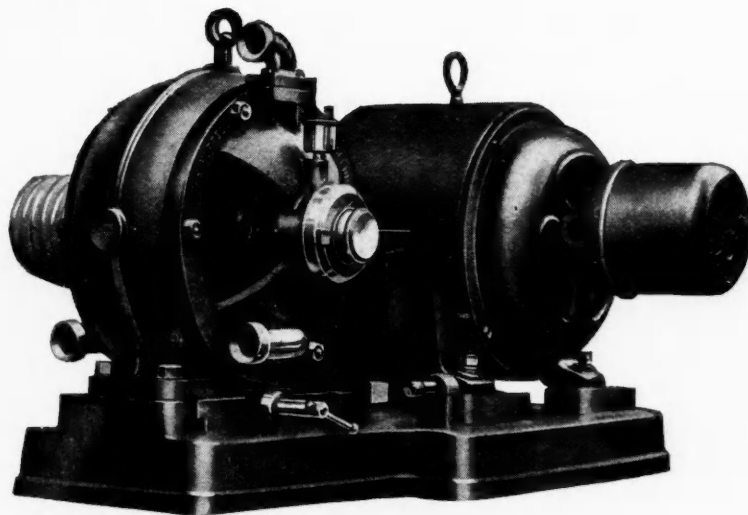
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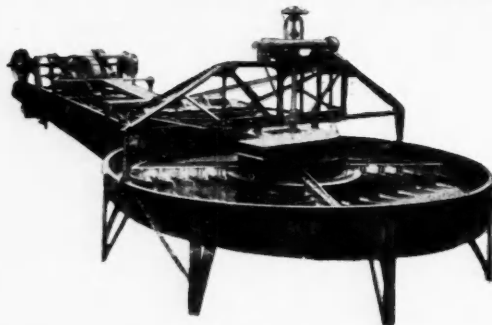
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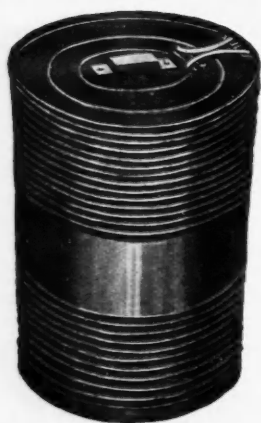
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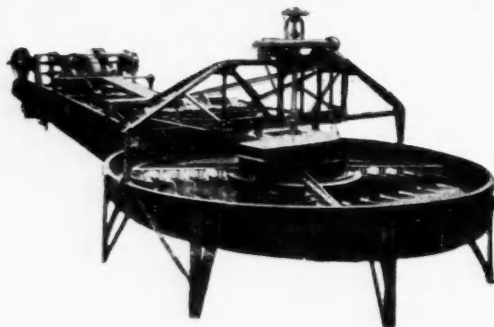
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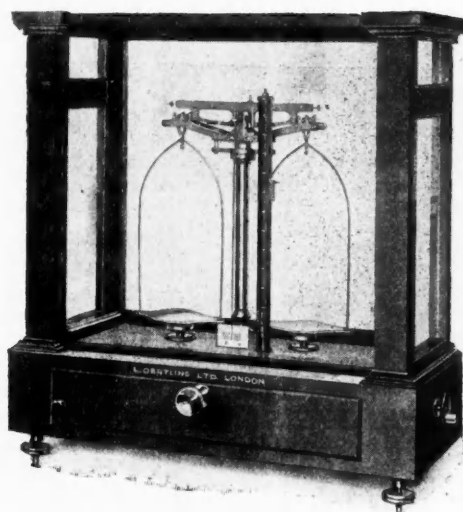
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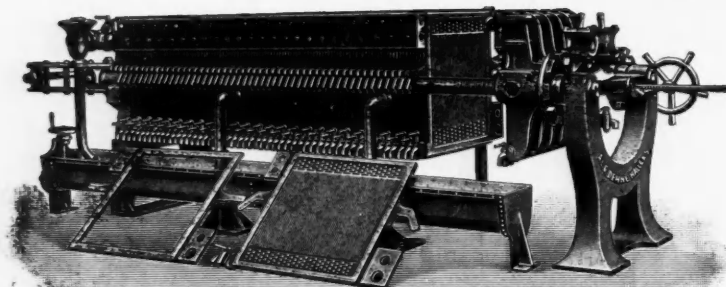
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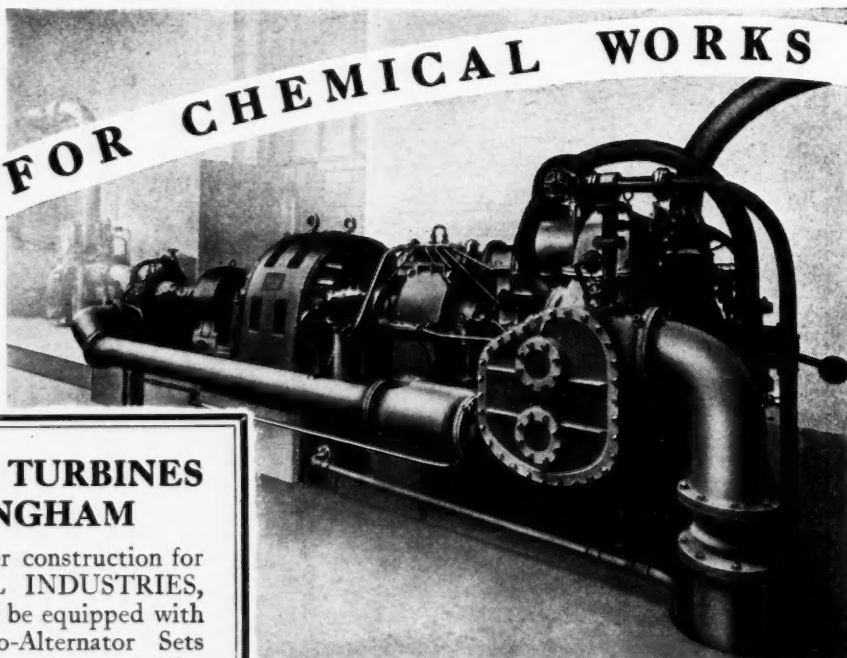
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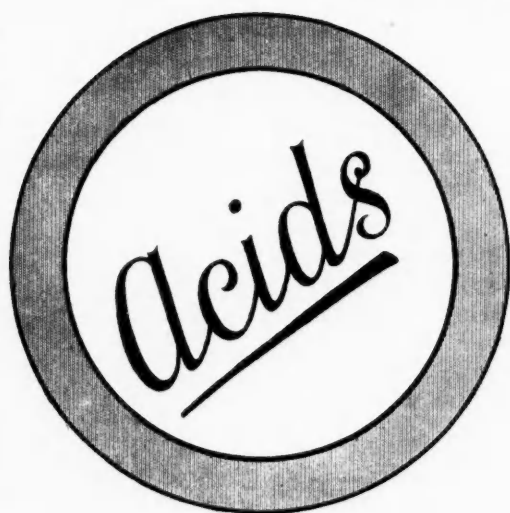
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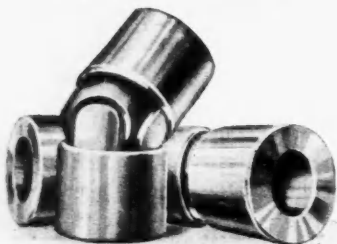


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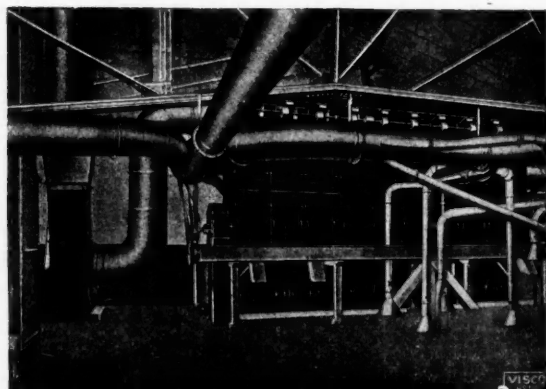
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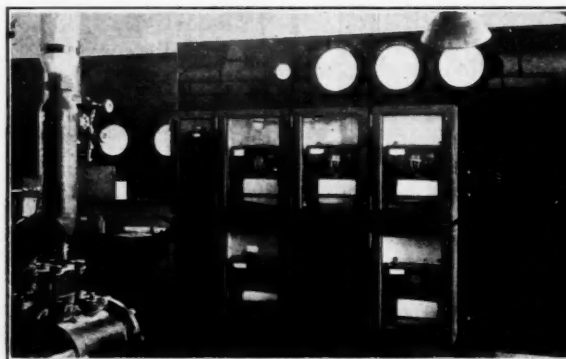
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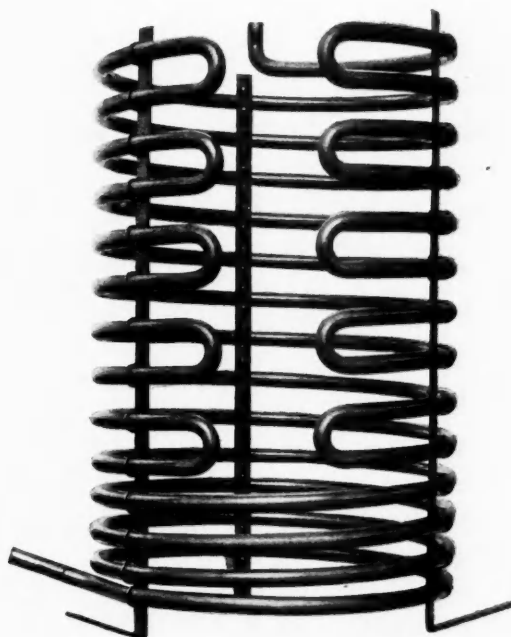
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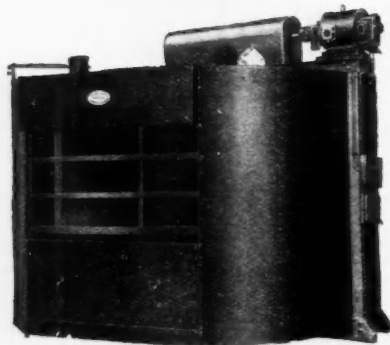
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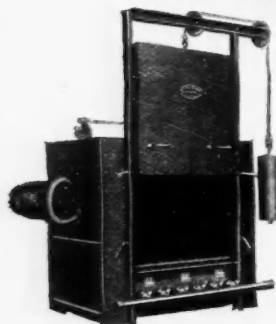
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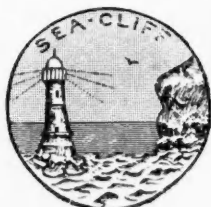
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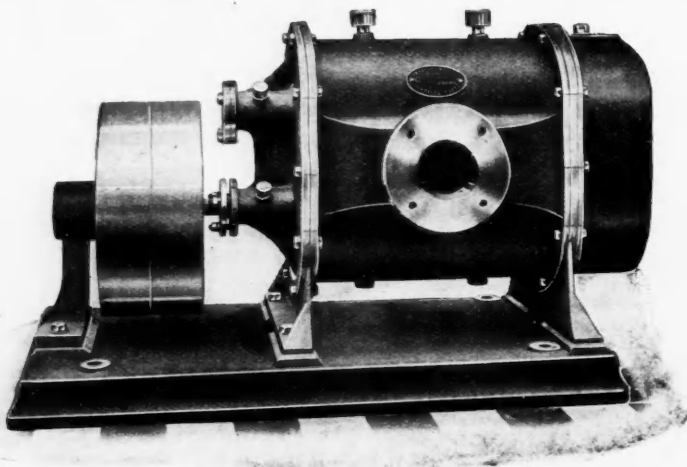
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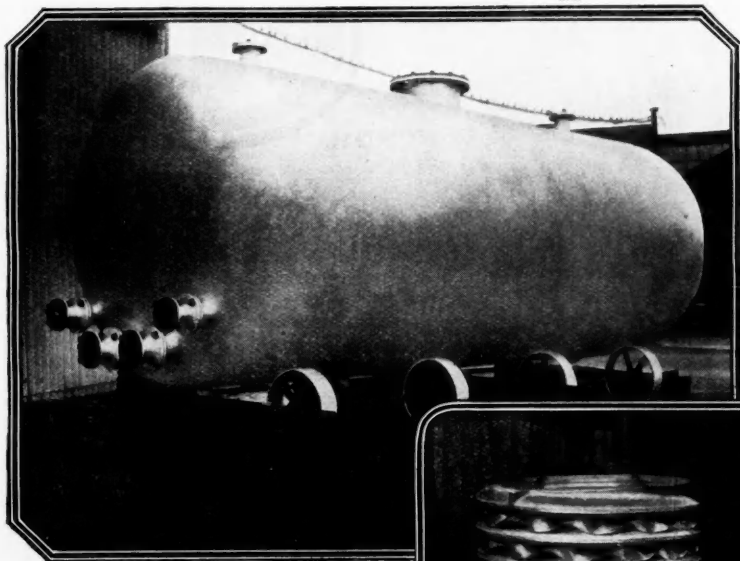
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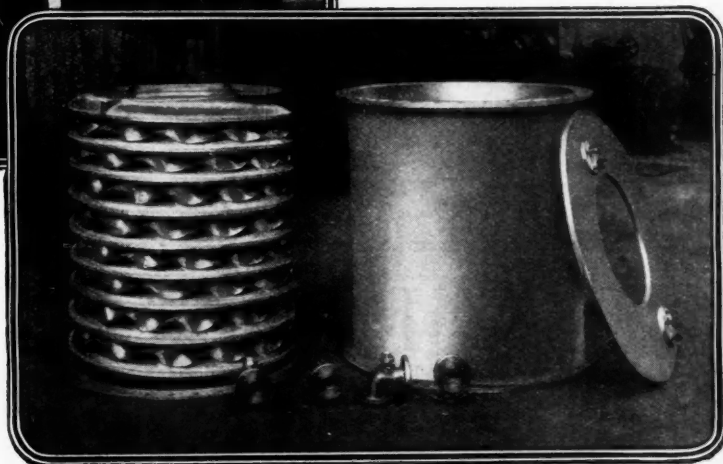
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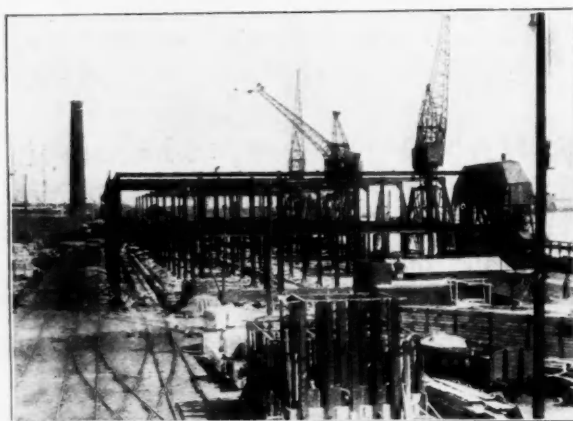
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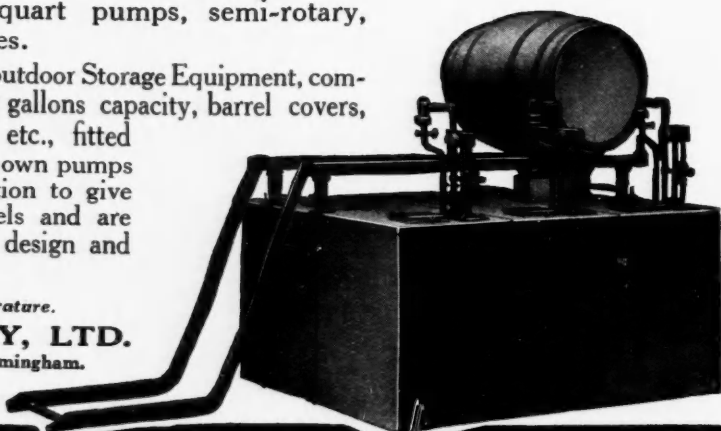
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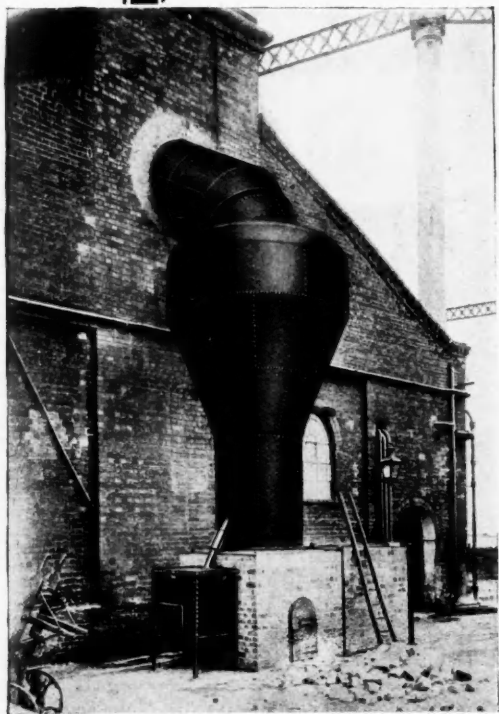
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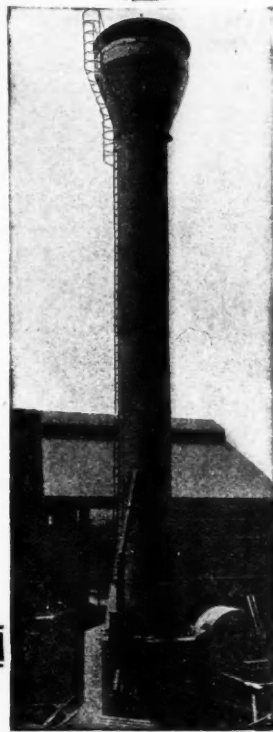
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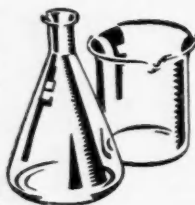
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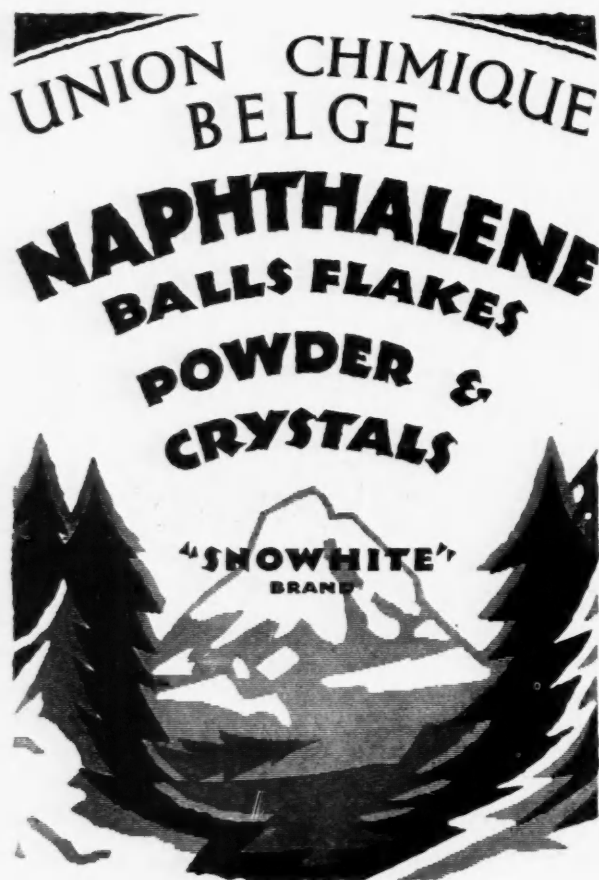
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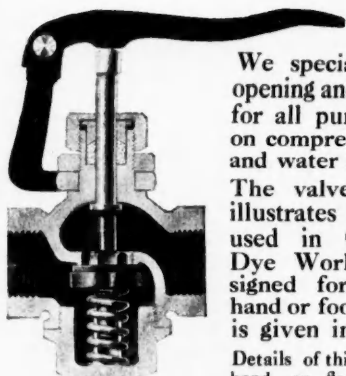
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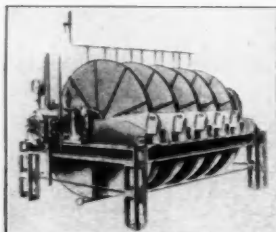
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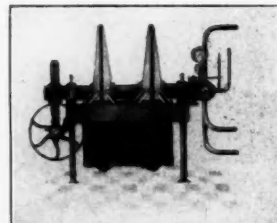
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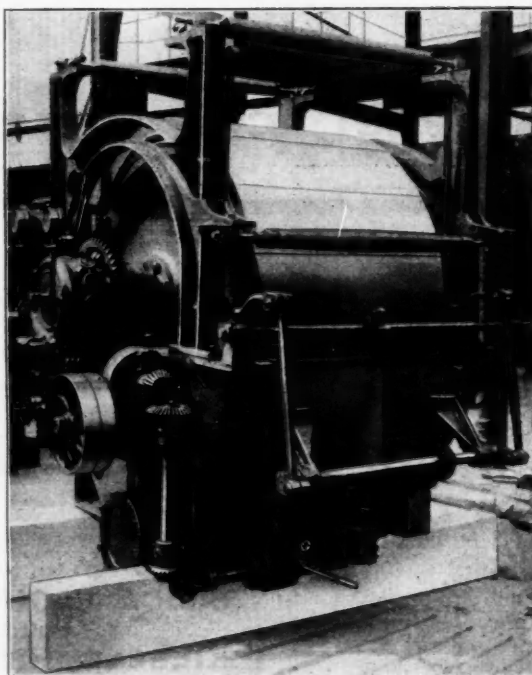
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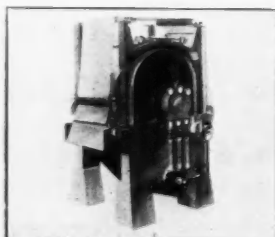
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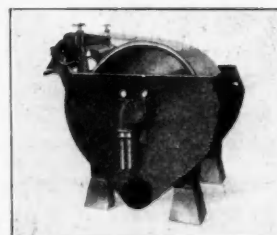
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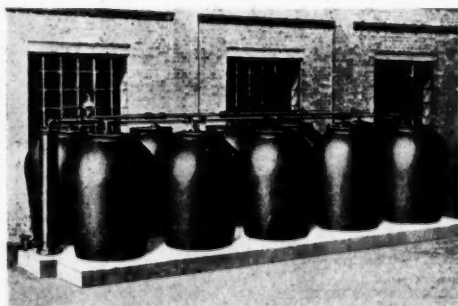
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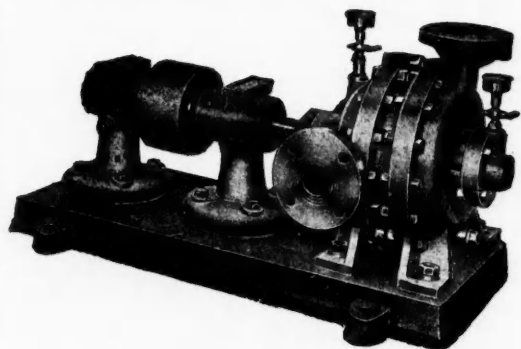
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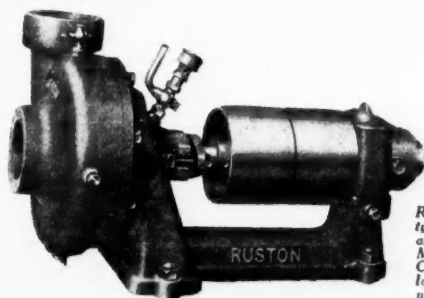
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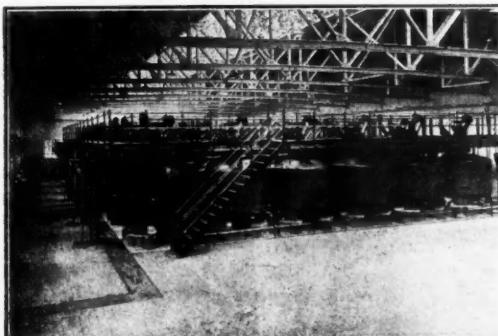
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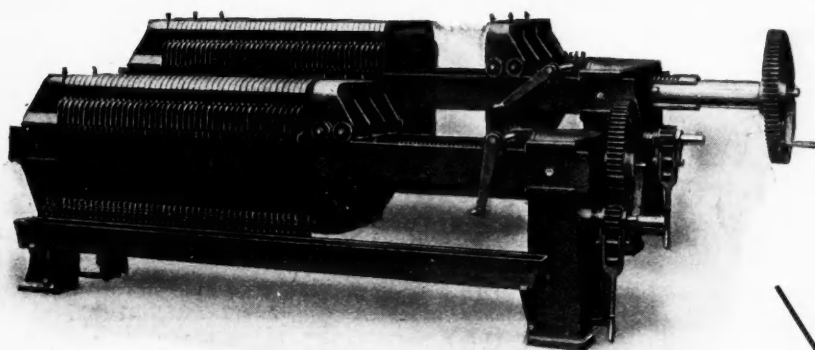
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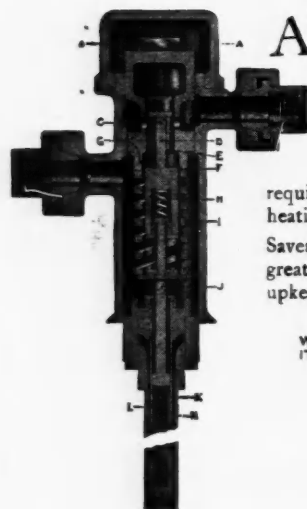
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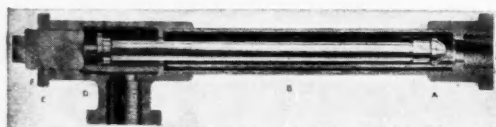
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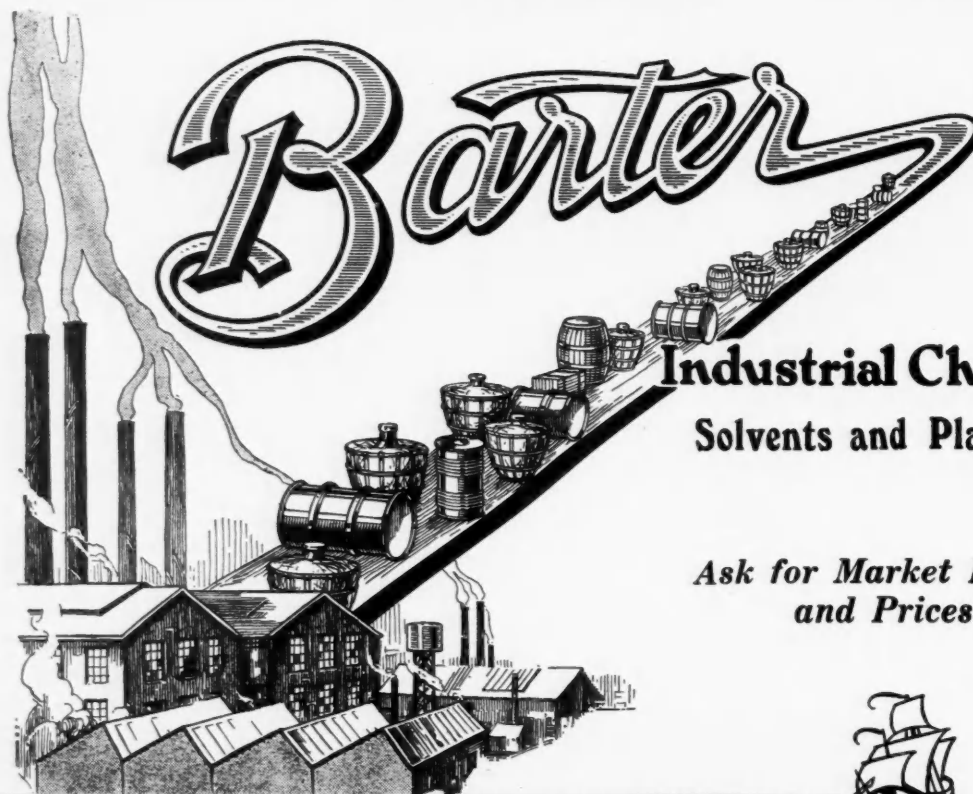
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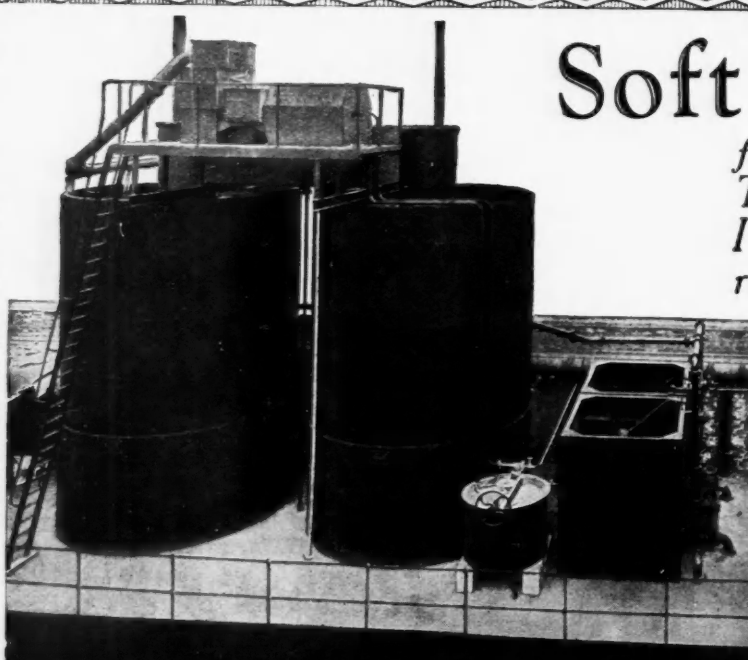
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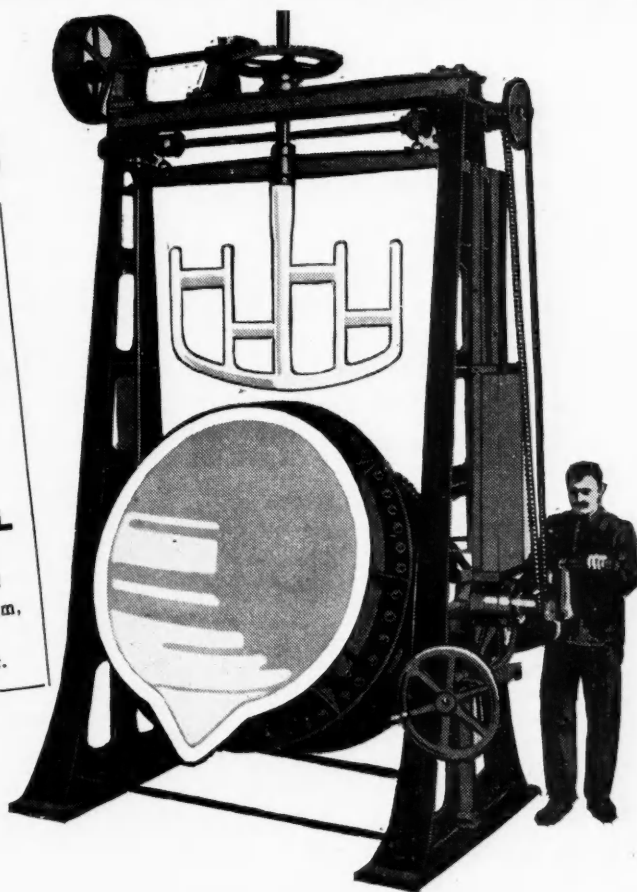
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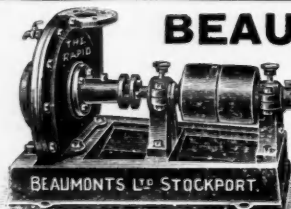
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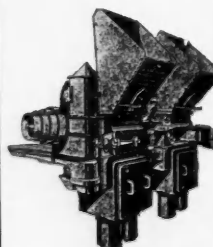
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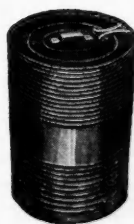
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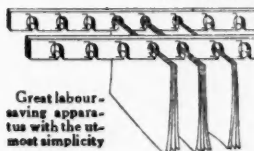
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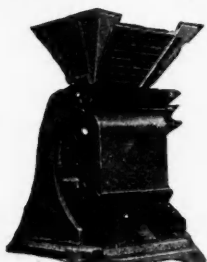
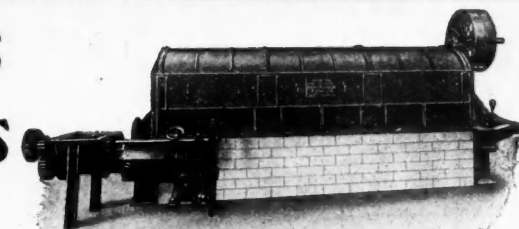
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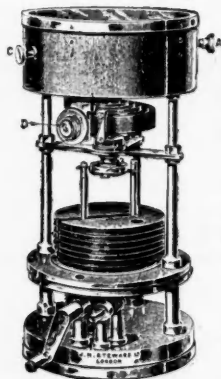
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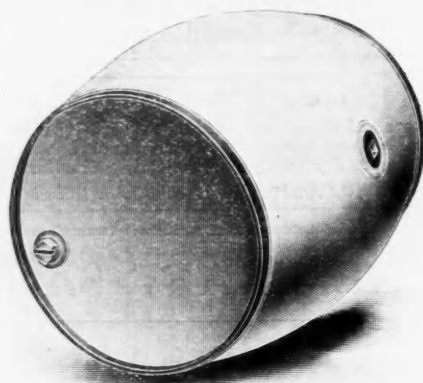
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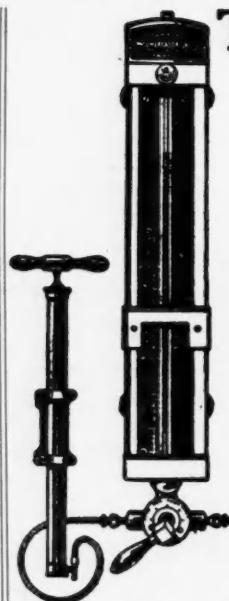
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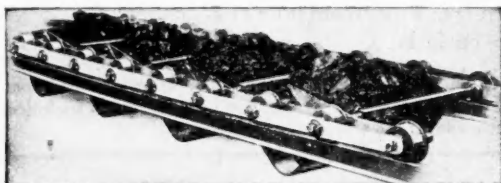
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